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Novel Dopants in Silica Based Fibers

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May 20, 1990

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## **I. Introduction**

The Laboratory for Lightwave Technology within the Division of Engineering at Brown University is one of the few university facilities in the U.S. with a capability to design and fabricate both optical fiber preforms, and then pull these preforms into fiber, and characterize the fiber. There are at present seven graduate students under my supervision, as well as four talented undergraduates. We are cooperating on several projects with professors from chemistry, physics, and various disciplines within the Division of Engineering. In addition, there is cooperative research with Northeastern University, several laboratories within AT&T (Murray Hill and Holmdel), and the Ruhr University, Bochum, Germany.

## **II. Research Accomplishments during 1989**

The main research accomplishment during 1989 was to develop our aerosol deposition technique to the point where we believe we have the best, most economical, and reliable method of fabricating fiber lasers. In principle, we have modified a K-Mart room humidifier as a source of aerosol for our MCVD doping process. Figure 1 and Table 1 are taken from a review paper by J. Simpson, of AT&T Bell Laboratory, Murry Hill, that was presented at the fall 1989 SPIE meeting in Boston, MA. In this first figure, Simpson briefly describes traditional doping techniques for placing substances whose compounds have a low vapor pressure into the preform. The rare earth elements all have inorganic compounds with high melting points, so traditional techniques using bubblers and oxygen as a carrier gas can not be used. Snitzer, while at Polaroid, used metal organic compounds containing rare earths to transport the dopant into the reaction zone in MCVD, but to do this, the whole passage way of the compounds used must be heated to several hundred degrees centigrade to insure that there is no condensation. As can be seen from Table 1, there are more advantages to the technique of aerosol doping that we have proposed than any other process. This is

due to the ability to structure the radial profile of the index of refraction. In addition, for reasons that will be discussed below, there is a high degree of axial uniformity.

In Figure 2, we see a schematic of our use of aerosol generation in conjunction with MCVD. The low vapor pressure of the inorganic compounds of, for example, the rare earth salts, precludes any vapor transport techniques. By dissolving our rare earth salts in water, they can be introduced into the ultra-sonic aerosol generator and then convected into the reaction zone. The aerosol size with our 1.5 MHz transducer is approximately four microns. The liquid from which the aerosol is formed is cooled to prevent agglomeration. In addition, thermophoretic forces keep the cooled aerosol particles away from the hotter wall. Indeed, one of the advantages of the aerosol deposition technique is that since energy must be put into the particles to vaporize them they are completely vaporized only when they reach the hot zone, especially if the region behind the torch is cooled. This guarantees that the reaction will not occur until the hot zone, thus leading to greater axial uniformity in the doping.

Our original work on aerosol doping was to transport rare earth elements, particularly Nd, in the form of a chloride dissolved in an aqueous solution. The aerosol droplet thus contained water, and  $\text{NdCl}_3$ . The water vaporized in the hot zone leaving a supmicron size particle of  $\text{NdCl}_3$ . This reacted with oxygen in the hot zone to produce the desired  $\text{Nd}_2\text{O}_3$ . Several preforms were made using this process, and by shining light down the preform, when observed from the side, it was possible to observe scattering. This indicated that there were sites of crystallization, albeit small, and that the losses from fiber drawn from this preform would not be acceptable. The preform was analyzed for us by Dr. Hong Po at the Polaroid Corporation and this is shown in Figure 3. The sharp spikes in the absorption

profile are indicative of crystalline sites. This was caused by the fact that the  $\text{Nd}_2\text{O}_3$  was not homogeneously distributed throughout the glass.

To circumvent this difficulty, we attempted to produce aerosol particles that contained (except for the oxygen) all of the constituents of the glass we wished to make. This guaranteed that there would be uniform distribution of the doping at the molecular level. The organometallic precursor that we found convenient to use as our solvent was TEOS (tetraethylorthosilane,  $\text{Si}(\text{OC}_2\text{H}_5)_4$ ). This is an inviscid, combustable liquid that can easily be formed into an aerosol. In addition, compounds of many dopants are soluble in it. For example, aluminum butoxide, a viscous liquid, the alkoxides or rare earth compounds, etc. Thus, our aerosol contains all of the components of the core glass. By December 1989, we had strong indication that this technique would solve any problems of crystallization that we had using inorganic precursors. Enclosed are two papers that were presented at the SPIE meeting in Boston.

In addition to the highly promising work on internal deposition using MCVD, we have also considered extending our work to OVD. This outside deposition process has the advantage of not using a substrate tube, thereby opening the possibility of soft glasses with higher nonlinear coefficients than silica. Our MCVD aerosol technique has a transport of 1 gm/min. This is more than adequate for core doping of preforms. However, for OVD a much higher mass transport rate is required. We have acquired an aerosol generator capable of transporting 100 gm/min of TEOS, which corresponds to 21 gm/min of silica. Since the droplet size is much larger, it can not be easily transported through a delivery system, and must be burned. We have performed some preliminary experiments on the combustion of TEOS and believe that this is a promising technique for the fabrication of gradient index lenses, soft glass fibers (high nonlinearity), and possibly in the fabrication

of substrate tubes of novel composition. We have also negotiated with AT&T Engineering Research Center, Princeton, NJ, for the donation of Dr. T. Miller's OVD laboratory.

### **III. Work Planned for the Future**

We will continue work on perfecting our MCVD aerosol process concentrating on fiber lasers, and will supply these fiber lasers to the optical computing group at AT&T Bell Laboratory, Holmdel, NJ. Much effort will also be devoted to getting our new OVD laboratory functional, so that we can make novel composition of glasses using organometallic precursors to form a bulk glass.

# LOW VAPOR PRESSURE DOPANT DELIVERY MCVD METHODS

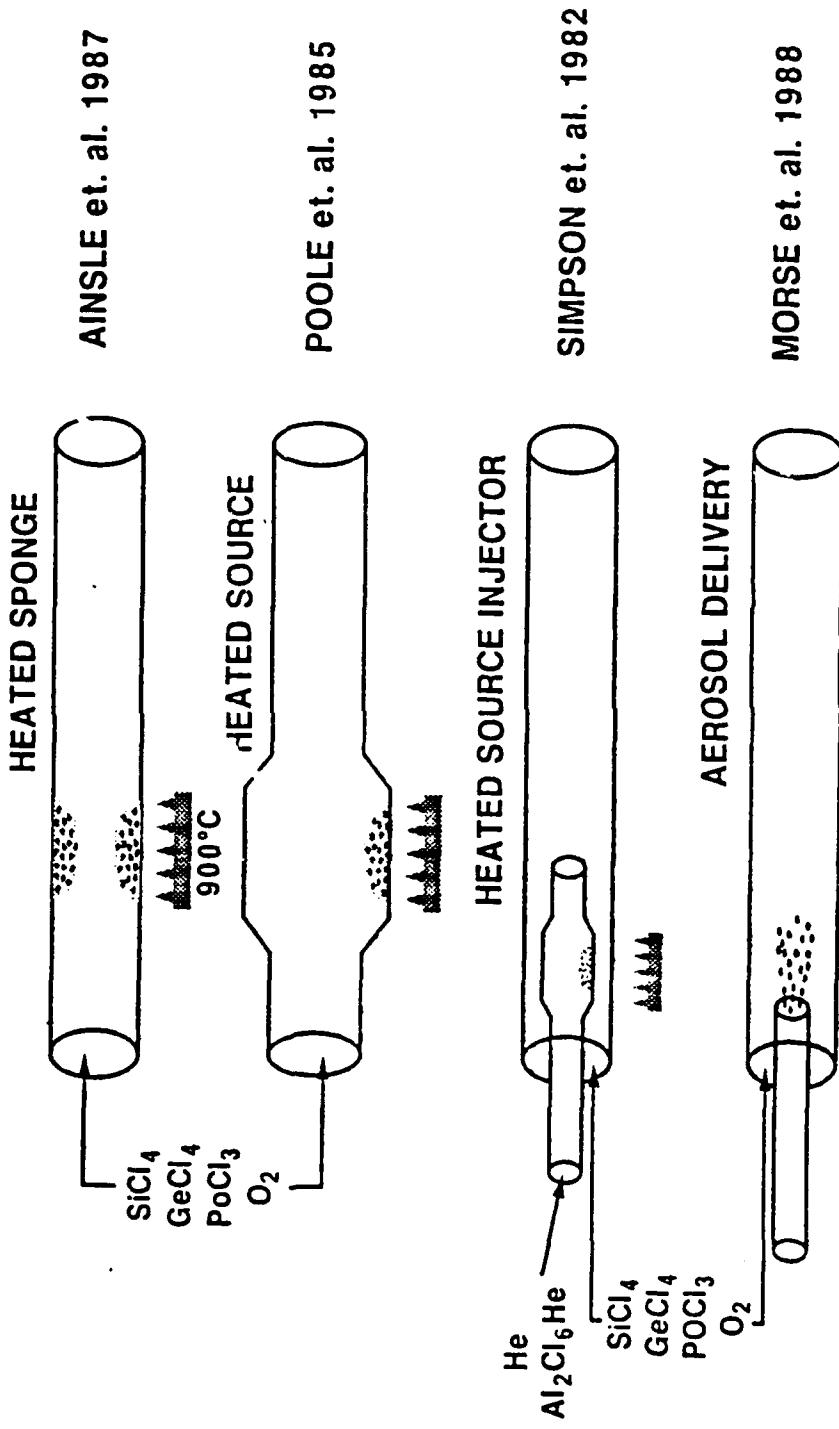


Figure 1. MCVD Fabrication Techniques for Fiber Lasers (J. Simpson, AT&T)

TABLE I.  
**TRADEOFF OF FABRICATION METHODS**

	Intrinsic Loss	Co-Dopant Concentration Control	Re-Dopant Profile Control	Alternate Hosts
<u>MCVD</u>				
VAPOR PHASE	↑	↓	↑	↓
SOLUTION	↓	↑	↓	↑
AEROSOL	↓	↑	↑	↑
<u>OVD/VAD</u>				
VAPOR PHASE	↑	↓	↓	↓
LIQUID PHASE	↓	↑	↑	↑

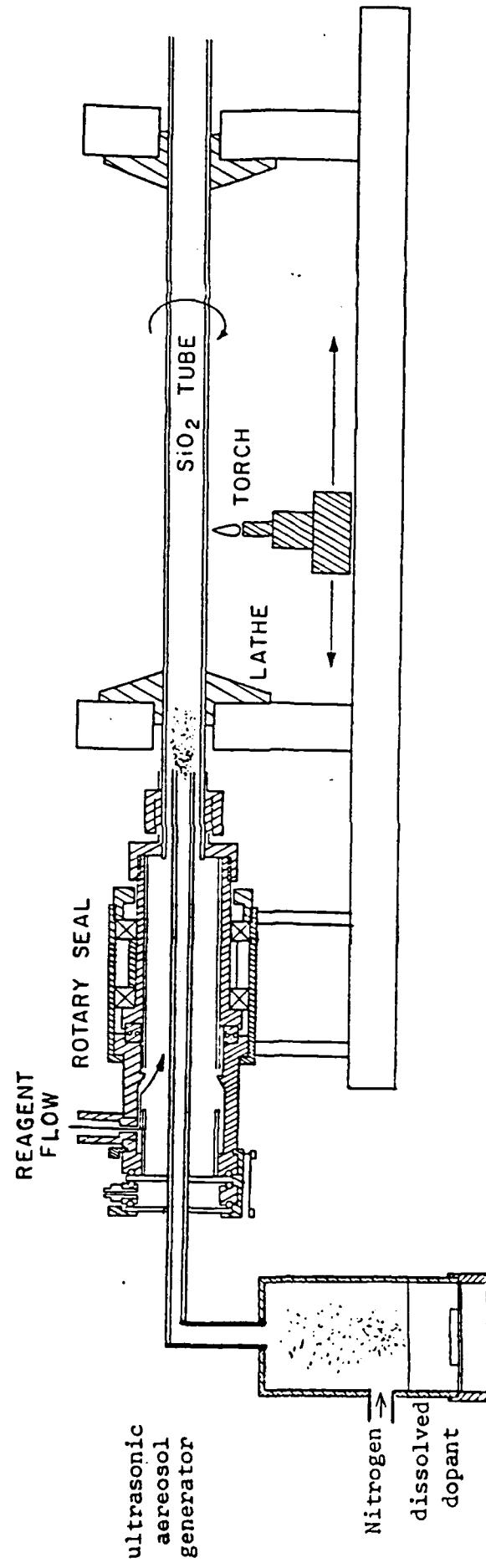
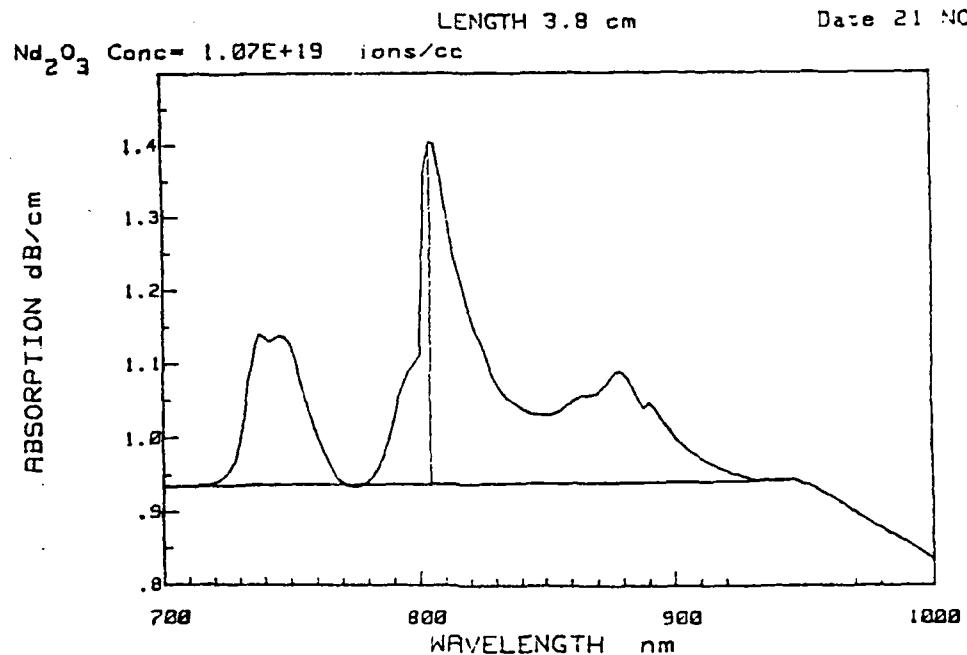
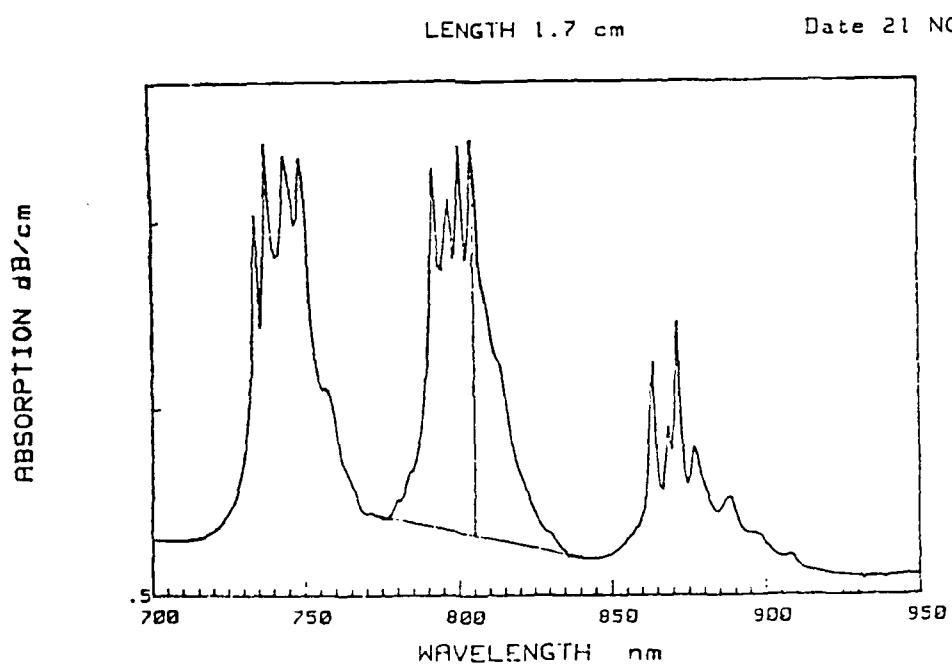


Figure 2. MCVD process with aerosol modification



a)



b)

Figure 3. Attenuation spectrum of Nd doped preform for:  
a) no crystallization, and b) crystallization

## Applications of Embedded Optical Fiber Sensors in Reinforced Concrete Buildings and Structures

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### ABSTRACT

The potential use of optical fiber sensors embedded, prior to curing, in reinforced concrete buildings and in structures such as bridges, dams and tanks is discussed with regards to the non-destructive measurement of internal strain, and the evaluation of structural integrity. Novel applications in the areas of structural monitoring, experimental stress analysis and in the management and control of service installations are presented. A discussion of the fundamental issues regarding the practical implementation of this technology is given. (R.H.)

### I. INTRODUCTION

The use of optical fiber sensors embedded in composite materials for the measurement of internal strain and detection of structural damage has recently been the subject of substantial research<sup>1</sup>. However, the focus of the applications is centered around the aerospace industry. In this paper, by contrast, we explore the possibilities of embedding optical fiber sensors, for the same purposes stated above, in buildings and structures such as water dams, bridges, nuclear reactors, or tanks, made of reinforced concrete.

Reinforced concrete engineering, although highly advanced in materials and in the structural analysis and design, is deficient in its evaluation techniques for concrete strength and structural integrity. Most of the methods used are destructive. Various nondestructive methods for testing concrete have been developed<sup>2</sup>. Such methods rely on the absorption, scattering and transmission of X-rays and Gamma rays by the concrete; its response to nuclear activation; its resistance to penetration by projectiles; or its resonant frequency. In a newer technique, iron particles are embedded into the concrete and stimulated with electromagnetic radiation<sup>3</sup>. Measurement of the sound waves or the surface temperatures produced yield information about the condition of the concrete and the structure.

Optical fibers, because of their small size, can be embedded within concrete without affecting its properties and used as sensible, but rugged, transducers of mechanical perturbations. In this respect, fibers have the ability to provide high resolution temperature and strain measurements, detect the onset and growth of cracks, as well as to monitor creep and thermal stresses.

Development of fiberoptic technologies for the civil engineering industry is attractive because the character of social service that most structures carry, along with the fact that building and heavy constructions in the United States alone amount to a 700 billion dollar business, with a weekly average of 6 billion dollars in new construction plans<sup>4</sup>. This offers the possibility of new fiberoptic markets, if fiber sensors can improve the evaluation of concrete by reducing testing and operation costs.

The basic physical principle behind embedded fibers to characterize the state of a composite material is that light sent through the fiber has either its intensity, phase, or polarization altered by changes in the mechanical state of the surrounding host. This can ultimately be used to provide "real time" information on the state of a specific concrete structure or member by means of a built-in damage detection and evaluation system based on a grid of optical fibers embedded within the structure at the time of construction.

Nondestructive, in-situ, testing of concrete properties will also be possible. For instance, its relative strength, its modulus of elasticity, or whether a batch was thoroughly mixed and properly poured and cured. Engineers and inspectors can return over the years to probe the internal condition of the concrete without damaging it., simply by connecting external electronic equipment to the connectorized ends of the embedded fiber sensor. This is particularly useful in buildings located in seismic regions, and in constructions which demand a high degree of safety and reliability as is the case with hospitals, auditoriums, bridges, dams, and others.

The fundamental applications envisioned for this new technology are listed in Table 1. These can be grouped in three main areas as follows: 1) structural monitoring and damage evaluation, 2) experimental stress analysis and, 3) management and control of systems and service installations in buildings. The first area studies the incorporation of single- and multi-mode fibers within structural concrete members such as beams, columns, arches or shells, so that concrete curing, stress, strain, deflection, bending, cracks and creep can be measured and monitored individually, as well as the deflection and bending of structures as a whole. In the field of experimental stress analysis, fibers would make sensitive and versatile sensors for the measurement of mechanical characteristics of structural members in experimental studies. The third set of applications considers that lighting, electric power, security, fire alarms and other building services can be run more efficiently and economically using fiber optic sensors to monitor the state of affairs of variables of interest (temperature, pressure, current, etc). Furthermore, integration of all the information supplied by the different fiber sensors into a single processing center within the same facility could result in a "smart" building.

In order to implement these ideas, a fundamental understanding of the fiber-host interaction is needed. This and other issues of relevance to the embedding of the fibers will be discussed in the following sections, along with a more detailed description of the applications.

## II. THEORY

- *Overview of Reinforced Concrete Fundamentals*

Reinforced concrete (RC) is probably today the most popular building material in the world because of its mechanical properties, and its architectural characteristics such as ease of use and of casting into different shapes. Simply put, RC is a composite material made of approximately 98% of concrete and the remaining 2% of reinforcing steel, by volume. Plain concrete consists of inert mineral aggregates (such as sand, stone and gravel) immersed in a matrix made from portland cement and water. The cement paste fills the voids between aggregate particles. Initially this mixture is plastic and workable but, as a result of an exothermic chemical reaction, the cement hardens to form a solid durable material. Concrete possesses a high compressive strength but it is weak in tension. So it has to be reinforced with steel bars, wires or welded fabric that absorb the tension loads in the structure, and thus the name RC.

There are several varieties of concrete<sup>5</sup> which have to do with the strength, weight, or length of curing time. However, by the type of application that it is destined for, concrete can be either formed or massive. The former is used for the casting of columns, beams, shells, slabs, etc.; whereas the latter, is present in the construction of water dams, piers, and foundations. Because of the high volumes associated with massive concrete some interesting problems arise during its curing, as we shall describe further on.

Once hardened, concrete starts developing considerable strength and continues to do so, although at a lower rate, for the remaining life of the structure. Strength is a function of water-cement ratio, mix characteristics and curing conditions (humidity and temperature). In general, compressive strengths for plain concrete are in the 35 to 50 MPa range. Tensile strength is deficient and about one tenth of the compressive one. Maximum unitary strains before failure in structural members have values of  $2 \times 10^{-3}$  to  $5 \times 10^{-3}$  mm/mm; however, confinement of the concrete by spirals or heavy ties, tend to increase these values. Average modulus of elasticity values are between 20 and 30 GPa.

Another important characteristic of concrete is its deformation with the moisture transport and variations in temperature. There are two types of moisture transport in concrete: shrinkage, which is caused by the contraction of the solid as it loses some of its water, partly by chemical reaction and partly by evaporation; and creep, which is caused by the squeezing of water out of the fine pores in concrete under sustained loads. Shrinkage of concrete ranges from  $2 \times 10^{-4}$  to  $5 \times 10^{-4}$  mm/mm. Temperature changes make concrete expand or contract introducing internal stresses if the movements are restrained. The thermal coefficient of expansion of hardened concrete is  $5 \times 10^{-6}/^{\circ}\text{C}$  which matches that of the reinforcing steel.

- *Optical Fiber Embedding in Concrete*

To perform the function of internal temperature/strain sensor, fibers must be embedded in the uncured concrete mix, and a good bonding must exist at the interface between fiber and cement host. This presents two major problems: a) the introduction, without damage, of the fibers into a plastic, aggregate-filled, and mechanically vibrated medium; and b) the chemical durability of the fibers in the water-rich, highly alkaline environment of a cement paste.

As seen from Fig. 1, which depicts a cut-out section of hardened concrete, about 70% of the volume is occupied by the aggregates. This means that there will be many obstacles in the way of a fiber in any given direction. Furthermore, fibers will be subjected to severe mechanical abuse as a consequence of the filling process of the concrete mix into the formwork which involves pumping, vibrating (to disperse the mix in hard to reach places) and shoveling. To overcome these difficulties we propose three different embedding techniques. In the first technique (Fig. 2a) a metallic protective tube surrounds the fiber. The tube is laid in place, and concrete poured on top of it. Once the filling operation is completed, the tube is slowly removed leaving the fiber in. This technique has the advantage of allowing the formation of a smooth cement boundary as a result of cement "bleeding" around the surface of the tube. The same effect occurs at the walls of the formwork and is the explanation for not seeing any aggregate marks on the exterior faces of concrete structures.

Fig. 2b illustrates a different approach where the fiber, along with two metallic ends attached to it, is allowed to move freely inside a metal sleeve. In this way, the internal forces are transmitted to the fiber without exposing it. The metallic encapsulation should be made out of a material with a coefficient of expansion matching that of concrete to minimize residual temperature-induced stresses. Lastly, in the third approach (Fig. 2c), naked fibers are held in place by securing them to the steel reinforcements by means of tape spacers. The spacers do not interfere with the concrete-fiber bonding.

Whatever the embedding technique employed, extreme care must be exercised during its deployment, or otherwise the fiber will be severely damaged and its use as a sensor element lost.

- *Chemical Attack*

Water and hydroxyl ions react with the surfaces of glass fibers reducing their strength due to the acceleration in the growth of cracks under stress. Water is only present in cement during the first few days of curing, since most of it is lost by chemical reaction or evaporation. Depending on its characteristics<sup>6</sup>, concrete can be highly impermeable, which explains why the reinforcing steel bars do not corrode inside the concrete.

On the other hand, cement is strongly alkaline (with a pH ~12) and will tend to corrode the glass fibers. Fortunately, considerable research has been done on the use of glass fibers as a reinforcing material in portland cement<sup>7</sup>. These studies have led to the development of alkali-resistant glass fibers<sup>8,9</sup> like the so-called Cem-Fil<sup>10</sup>, characterized by having about 17% by weight of zirconia ( $\text{ZrO}_2$ ) in its composition. Experimentally it has been demonstrated that polymeric coatings on glass fibers improve their chemical durability allowing the use of non-alkaline fibers<sup>11,12</sup>. In particular, optical silica glass fibers with high alkaline resistance were obtained by coating them with polycetherimide<sup>13</sup>.

- *Interface Bonding*

An adequate bond is obtained between concrete and fibers by virtue of the shrinkage that takes place during curing, caused by the loss of moisture with a decrease in the volume of the mix of about 0.05 percent. In addition, there will be some mechanical keying and constriction caused by the crystal growth of the hydrated calcium alumino silicates. This latter effect might be also detrimental to the performance of the fibers, so alkali-resistant coatings with an adequate compliance should be used to protect the fibers.

### III. APPLICATIONS

- *Structural Monitoring and Damage Evaluation*

#### Thermal stresses and curing

Temperature gradients within a concrete structure arise from cement hydration during curing, or changes in ambient temperature (freezing and thawing, wetting and drying, or heating and cooling). These temperature differences give rise to differential volume changes. When the tensile strains from these volume changes exceed the tensile strain capacity of concrete, cracks will appear. Deflection and rotation of structural members are another consequence of temperature changes.

The effects of cement hydration are not critical in regular cast concrete structures, but it is a major consideration in massive concrete members. Curing of concrete is always accompanied by liberation of heat from the chemical reactions which bring about the hardening of the portland cement. This heat of hydration, as it is called, causes the internal temperature of concrete to rise, on the order of 10 F per 100 lb of cement, in contrast to the exterior surfaces. As cooling begins, the outermost layers rapidly gain both strength and stiffness. Any restraint of the fine contractions will result in tensile stresses. Evidently, large amounts of heat need to be dissipated adequately during the curing process if flawless concrete is desired. In this respect, embedded fiber optic temperature sensors would make possible the measurement of the internal temperatures in massive concrete, allowing to control the concrete cooling rate. The distribution of temperature along a conventional silica fiber can be obtained by measuring the temperature dependent Raman backscattering in the fiber<sup>14</sup>. This is accomplished by filtering out the antistokes signal component from an OTDR signature.

A set of several temperature sensors would facilitate the analysis of the heat transfer between cured and uncured regions. Furthermore, a network composed of several temperature sensors situated in strategic members all around the structure would supply information on the building's thermal stress behavior year round and on the dynamics of the heat transfer due to temperature changes from day to night or from summer to winter.

#### Cracks and defects

The presence of cracks in RC is inevitable. Cracks are produced by the reinforcing steel being stressed under normal load conditions, restrained drying shrinkage, or temperature-induced volumetric changes. The significance of cracks varies, they may represent the total extent of the damage or even point to problems of greater magnitude. Nonetheless, the safety of the structures depend on keeping the width of cracks, located in structurally critical places, below a permissible maximum.

The onset and growth of cracks in concrete can easily be detected by monitoring the light continuity in single- or multi-mode fibers strategically embedded. In this way, the transmitted light intensity through the fiber will be sharply reduced or totally interrupted if the fiber is severed by excessive strain from a crack. The location of the break can be determined by interrogating the back-reflected signal using OTDR or OFDR techniques. The same principles can be applied to determine the presence and location of reinforcement bars, voids, flaws, honeycombing, or density changes in the concrete that otherwise are not detectable by visual inspection.

## Strength

Because the direct determination of concrete strength implies the loading of specimens up to failure, it becomes attractive the use of non-destructive methods. Although such techniques cannot be expected to yield absolute values of strength, they allow for the comparative testing of cured concrete on-site. A number of different devices and techniques have been developed<sup>2</sup> all of which measure some other property of concrete from which an estimate of its strength, durability and elastic modulus can be obtained.

Optical fibers can be used as a non-destructive, invasive, strength measuring sensor. Two different schemes are envisioned for this purpose. Fig. 3 shows a single-mode fiber embedded a few centimeters from the surface. Mechanical pressure is then applied onto the fiber by means of a crank screw attached on the surface of the concrete. As the applied pressure increases, the amount of transmitted light through the fiber decreases accordingly and should vary as a function of the concrete's modulus or strength. In the second method(see Fig. 4), a birefringent fiber is buried deep into the concrete. The output of said fiber is connected to a polarimeter to detect the changes in the state of polarization of the outgoing light as a consequence of strain perturbations produced by stress waves travelling normal to the fiber's axis, and artificially generated by means of an external hammer or an electro-mechanical transducer. Measurement of the time interval that elapses between the polarization changes in two consecutive fibers, a known distance apart, can lead to the determination of the so-called "pulse velocity" in the medium. This velocity is almost independent of the geometry of the material, but strongly dependent on its elastic properties, hence it may be used as a measure of strength provided and adequate relationship between pulse velocity and strength has been determined.

## Bending and deflection

Other mechanical phenomena of interest to civil engineers and architects are the deflection and bending of long-span members and frames. In this case it is possible to use external fiber sensors rather than embedded ones. For instance, a fiber microbend strain sensor for detecting bending and vibrations of large mechanical structures has been devised and tested<sup>15</sup>. Bending radii is related to a change in transmitted light intensity for a fiber placed between two corrugated pieces that induce microbending losses on it as they move relative to each other under flexure. Furthermore, many sensors attached to different parts of the structure can be monitored simultaneously by connecting them in tandem along a single fiber. Identification of each sensor is possible from its position in the OTDR signature.

Similarly, deflections can be determined using a fiber optic strain gauge<sup>16</sup>. In this arrangement two fibers of the same length are attached to the top and bottom surfaces of the member under study, say a beam. As the beam deflects, one fiber will be longitudinally stretched, while the other will be compressed. The different strains in the two fibers cause a difference in optical path lengths which results in an interference pattern of the output light beams. The deflection (or strain) in the element can be calculated from the motion of the interference fringes.

## Strain, stress and shear

A RC structure is a continuous frame in which all members are rigidly connected to one another. The loads are therefore supported not only by the structural member on which they are acting, but also by the members to which it is connected. Said members are deformed under the action of tensile and compressive forces produced by the loads that the structure must bear. If the acting loads are within the designed range, the deformations will be elastic. However, if exceeded, plastic deformations and even failure will occur. Associated with these deformations are internal stresses and strains that differ in character and intensity depending upon their location in the structure, and on the nature of the loads. However, knowledge of the stress/strain state of the structure is vital for the prediction of its behavior. Mechanical strains, from which stresses can subsequently be inferred, can be determined with relative ease by embedding fibers within critical concrete members. Dimensional and/or refractive index changes in the fiber can be used to measure the strain exerted on the fiber. Experimentally, minimum detectable strains as low as  $10^{-6}$  mm/mm have been demonstrated<sup>15</sup>. This is at least two orders of magnitude smaller than the minimum strain present in concrete. Different techniques for strain measurement have been developed<sup>17-21</sup>. These techniques make use of either single- or twin-core fibers.

The twin-core fiber strain sensor, developed by United Technologies Research Center<sup>17</sup>, consists of a fiber with two matched, closely spaced, singlemode cores. When coherent light is launched into one of the cores, symmetric and antisymmetric modes, having different phase velocities, are excited. These two modes exhibit a periodic interference along the length of the fiber which results in the back and forth coupling of light from one core to another. External perturbations such as temperature and strain influence the characteristics of the crosstalk between cores. Operation at two different wavelengths allows the simultaneous determination of temperature and strain<sup>18</sup>.

Single-core fiber sensors, on the other hand, rely on changes of either polarization, phase, intensity, backscatter or mode coupling to measure strain. One of the simplest configurations is the polarimetric sensor<sup>19</sup>. It consists of a singlemode birefringent fiber into which polarized light is launched. Mechanical perturbations are sensed by measuring the polarization coupling between the two orthogonal, linearly polarized, modes travelling through the core. Moreover, it can be used to measure either transversal (normal to the fiber axis) or longitudinal (along the fiber) strains, as well as vibrations.

A common type of fiberoptic strain sensor is the Mach-Zehnder interferometer<sup>20</sup>. Where two singlemode fibers act as the arms of a MZ interferometer. The sensing fiber is embedded or attached to the structure while the other fiber is isolated from it. Strain is determined by measuring the phase difference between both fibers.

Another class of sensor makes use of a singlemode fiber operating in the few-mode regime<sup>21</sup>. Strain is determined from the power transfer between two or more modes travelling in the fiber.

Distributed strain sensors can be achieved by interrogating a fiber along its length using Optical Time Domain Reflectometry (OTDR). In this technique, a short pulse of light is launched into an optical fiber. At each point in the fiber, there is some amount of backscatter from this pulse due to internal imperfections and external perturbations. This backscattered signal can provide information on the location and intensity of the parameters of interest (temperature and strain in this case). In a variation of this technique, fusion splices are made at known intervals along the fiber. These splices act as backscatter signal markers. Any elongation produced by strain on the fiber will affect the return time of the backreflected pulses.

Adequate placement of the fiberoptic strain sensors is crucial for their effective use. Because concrete is strong in compression, but weak in tension, tensile stresses are of more concern and where attention is focussed (depending on the type of structural member and load nature). Thus, optical fiber strain sensors should be embedded parallel to the steel reinforcement, where the tension forces will be present. In RC beams, in particular, the tensile stresses in the elastic regime are proportional to the distance from the neutral axis, being zero right at the axis. How the stresses vary and where the neutral axis actually lies, depend on the type of beam, the amount of load, and on the history of past loadings. This tensile stress/strain gradient can be determined by embedding fibers at different distances from the neutral axis.

Fibers should also be embedded next to the stirrups, which are steel bars for the reinforcement of diagonal tensile stresses resulting from the interaction between horizontal and vertical shear stresses.

- ***Experimental Stress Analysis***

Sometimes structures and structural members are of a shape so complex that it is difficult to solve them by a theoretical analysis. An answer can be obtained by measuring the stresses in some experimental way. In this context, fibers would make a far more sensitive and versatile sensor than conventional strain gauges. Fibers could also be used to better understand the behavior and failure of certain members. For instance, a grid of fibers could be embedded in the runways of airports to evaluate the state of stress on the pavement during the landing and take-off of airplanes. A 2-D strain mapping would be obtained that would help redesign and maintain such pavements.

Furthermore, embedded fiber optic strain sensors would enable engineers to compare between measured and designed values of bending moments and deflections. From this information correction factors can be determined that would make the structure more economical and/or safer.

- *Management and Control of Building Systems and Service Installations*

Advanced telecommunications and computer technology have revolutionized the office environment. Modern offices today accommodate several computer terminals as well as communication and text processing equipment, causing cable routing and increased heat generation problems. New office buildings have a tendency to grow vertically to get maximum use of the built area. However, the number of services with their associated hardware and wiring have increased accordingly becoming some sort of "high-tech jungles". Furthermore, it is desirable to reduce a building's operation and maintenance costs. In order to cope with such new trend a new concept in building engineering has been developed: The Integrated Building<sup>22-24</sup>, where all the building's main services are supervised and controlled by a centralized computer system.

Two major problems are in the way of implementing such concept: a) adequate communication links and networks with both flexibility and high capacity for the transmission of a dense flux of information, and b) the development of adequate sensor systems for the collection of data from the different building's services. Optical fiber networks and sensors are an ideal solution to these problems thanks to their characteristics such as small size, light weight, EMI immunity, high capacity, etc. Fiber-optic-based local area networks and other links have already found their way into office buildings, but the idea of using the fiber both as sensor and transmission medium in such environments is completely new.

Basic building services for which sensor systems need to be developed include: heating, air conditioning, sanitation, electrical power, lighting, elevators, fire alarms and security access control. However, most of the requirements could be fulfilled by simple temperature, pressure and current optical fiber sensors. Use of these sensors would make possible to measure the pressure, flow and temperature of water pipes to regulate the temperature and distribution of water supplies by controlling electrovalves, pumps and boilers as required. In a similar way, temperature in rooms, hallways and corridors could be monitored and controlled automatically. Electric current demands could be continuously monitored so as to redistribute loads, correct power factors or take action during overloads. Lights could be turned on and off according to the time of day. Fibers coated with special heat-sensitive polymers could be used as distributed heat sensors for fire alarm systems. And the list goes on.

As illustrated in Fig. 5 the outputs from the service installation and the structurally embedded sensors can be linked together and connected to other higher level services, such as telecommunications and office automation, through a central computer system which acts as a Building Command Center. This would result in a "smart" building, or possibly even a structure, capable of being interrogated and controlled by a single operator. Although these ideas probably sound to futuristic, they may well prove to be the way to go in the field of building engineering.

#### IV. SUMMARY

Fiber optic sensing technology offers the possibility of nondestructive, in-situ, measurement of the temperature, strain, stress and deformation of structures made of reinforced concrete. Novel applications for concrete buildings are envisioned in the analysis and evaluation of structural integrity, and in the management and control of building's services. However, in order to achieve this ultimate goal, experimental research is needed to investigate the feasibility of the ideas outlined in this paper. From this discussion, it can be noted that there are several important and interesting aspects on this subject that we hope will enhance the field of fiber optic sensors and smart skins.

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Table 1. Applications of embedded fibers in concrete

• CONCRETE

strength  
modulus of elasticity  
shrinkage  
curing  
uniformity  
voids and defects

• STRUCTURAL

thermal stresses  
structural monitoring  
damage evaluation  
strain  
stress  
shear  
bending  
deflection  
heat transfer dynamics

• SERVICE INSTALLATIONS

telecommunications  
office automation  
temperature, pressure, electric current

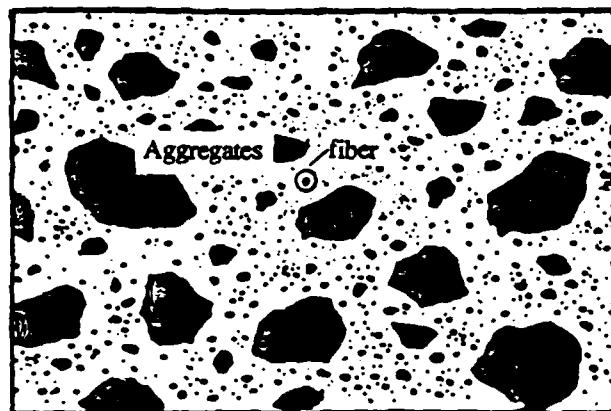


Figure 1. Cut-out section of hardened concrete

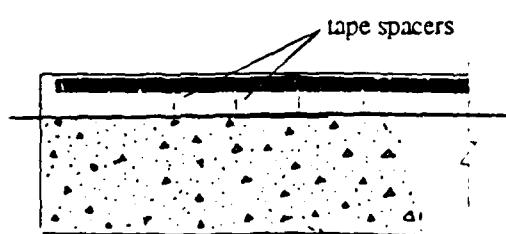
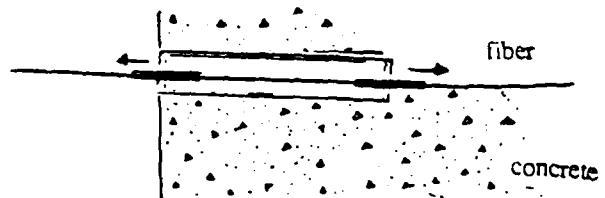
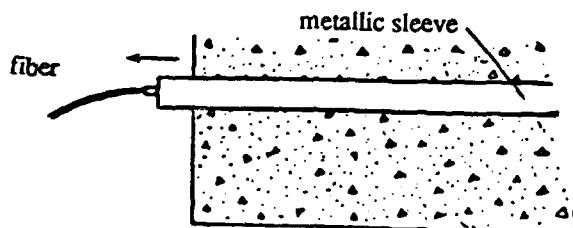


Figure 2. Proposed embedding techniques

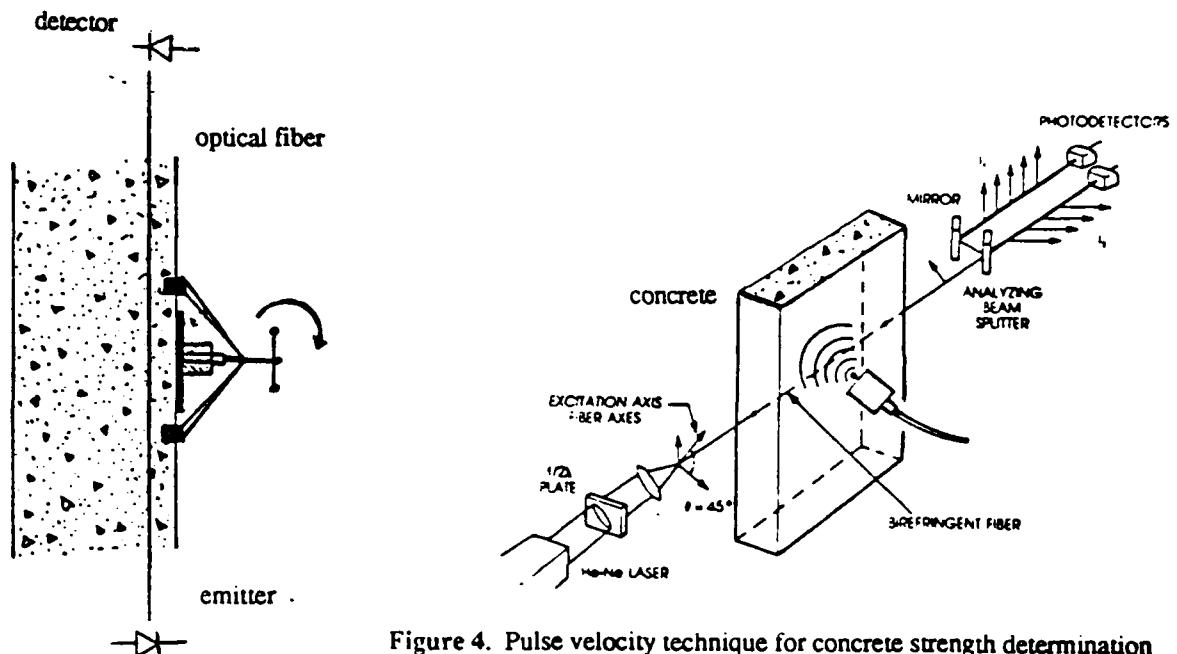


Figure 4. Pulse velocity technique for concrete strength determination

Figure 3. Determination of concrete strength by fiber compression

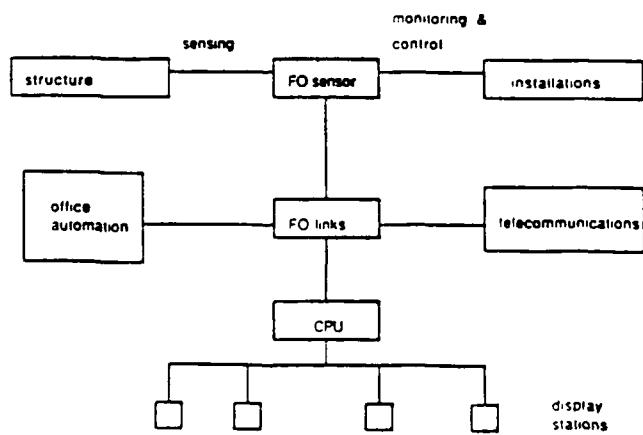


Figure 5. Block diagram of a "smart" building

## OVERVIEW OF FIBER OPTIC RESEARCH AT BROWN UNIVERSITY

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### ABSTRACT

Research in the Laboratory for Lightwave Technology is focused on novel applications of specially designed optical fibers. Emphasis is placed on new techniques of dopant incorporation, using both MCVD (Modified Chemical Vapor Deposition) and OVD (Outside Vapor Deposition) to fabricate fibers that are primarily for non-telecommunications applications. The topics pursued are fibers for fiber lasers, nonlinear effects in fibers, in particular, second harmonic generation, fiber devices for the measurement of electric field, new techniques for the fabrication of bulk-glass gradient index lenses, fiber designs for embedded fibers for sensor elements of future "smart skin" composites, the poling of glasses to induce anisotropy or possibly optical activity, and finally, fiber designs for N x N fused taper couplers.

### 1. INTRODUCTION

A Laboratory for Lightwave Technology has been established within the Division of Engineering at Brown University. This facility consists of a deposition laboratory for both MCVD (Modified Chemical Vapor Deposition) and OVD (Outside Vapor Deposition),<sup>1-6</sup> a fiber and preform characterization laboratory, a 7.8 m optical fiber draw tower, and a computer controlled station for the fabrication of fused taper couplers, a recent acquisition from the Polaroid Corporation. Brown is one of the few U.S. universities at which it is possible to design and fabricate novel optical fibers for sensor and other opto-electronic applications. This equipment is supplemented by NMR and Raman equipment for bulk characterization of inorganic glasses in the Departments of Physics and Chemistry. The support for the equipment in this laboratory has come primarily from the Department of Defense, the National Science Foundation, and from equipment gifts from industry (AT&T, Bellcore, the Polaroid Corporation). Research is being carried out in cooperation with faculty from the Department of Chemistry, Physics, and also with faculty and staff from electrical and materials engineering. Prof. W. Risen, Jr., of the Brown University Department of Chemistry, is carrying out a program, reported on here, on rare earth sol-gel glasses. In addition, cooperative research on second harmonic generation<sup>7-21</sup> in fibers is in progress with Prof. Ulf Österberg of the USAF Frank Seiler Research Laboratory, and Prof. Nabil Lawandy, Division of Engineering, Brown University. We are also collaborating on some novel concepts of "soot" deposition with Prof. M. Fiebig of the Ruhr Universität, Bochum, Germany. All of the modelling work on the thermal processes has been done in conjunction with Prof. John Cipolla, Jr., of the Northeastern University Department of Mechanical Engineering. Research in embedded fibers, and stress effects in fibers is done in conjunction with Prof. L.B. Freund and Prof. K.S. Kim of Brown University.

Our research program emphasizes new techniques of preform fabrication, the study of specially doped preforms for use as sensors, fiber lasers, polarization maintaining fibers, studies of stress induced effects in fibers, and nonlinear phenomena in fibers. We also use NMR, ESR and Raman techniques to study incorporation of novel as well as traditional dopants and how processing affects the manner in which these dopants are incorporated into a glass matrix. Emphasis is placed not on the traditional telecommunications fiber, but on special fibers and fibers associated with sensor applications.

The use of specialty optical fibers as sensors in such disparate fields as solid mechanics, chemical species detection, biology and medicine, creates a seemingly endless list of possible areas of application. We are clearly not in a position to pursue active research into all of the areas in which we believe optical fibers will play an increasingly important role. Our main thrust is to develop a fundamental understanding of the material and structural attributes of specialty fibers and to apply these in a host of areas we believe of importance.

## 2. RESEARCH AREAS

### 2.1 Aerosol Deposition in MCVD, OVD

Aerosol techniques are being developed for the incorporation of low vapor pressure dopants in MCVD, and for high mass flow formation of "soft glasses" using OVD for nonlinear fibers, and for GRIN (Gradient Index Lenses). For the core doping in MCVD, we have employed water, alcohol,  $\text{SiCl}_4$ , and various organic liquids as solvents for both organic and inorganic compounds of the rare earth elements. These solutions have been nebulized with a transducer from a modified "room humidifier" that produces 1 gm/min of aerosol. The aerosol is transported through a rotating seal in MCVD into the reaction zone where the desired oxide is produced. Uniformity of axial deposition has been achieved, and this technique seems suitable for the incorporation of many low vapor pressure compounds into the glass structure of a preform.

There is increasing interest in fibers that exhibit a high value of  $\chi^{(3)}$ , both for possible optical fiber switches in optical computers with a linear architecture, and for fibers that can efficiently produce the second harmonic. Many of these glasses have lower formation temperatures than silica, and the high collapse temperature of an MCVD substrate tube indicates the suitability of OVD for "soft glass" deposition. To this end, a high mass flow rate aerosol burner has been developed, and we have had some initial success with organic precursors to form a sodium, barium, silicate glass. Further details of this aspect of our program are presented in a companion paper in this conference on "Aerosol Doping Technique for MCVD and OVD".

### 2.2 Computer Modelling of Heat and Mass Transfer in Fiber Fabrication

This work is being carried out in conjunction with Prof. J. Cipolla, of the Mechanical Engineering Department, Northeastern University. In our attempt to increase efficiencies of the fabrication process for optical materials, a computational effort complements our experimental program. This involves the solution of the energy equation in conjunction with the equations of mass transport, momentum, and diffusion. Thus far we have examined the problem of dopant transport during the sintering and collapse phase of the fabrication of MCVD preforms, and have, we believe, an explanation of the "wiggles" that occur in a typical phosphorous-silica cladding, and how the concentration of dopant in the background gas is the dominant mechanism in determining dopant incorporation in each individual MCVD layer.<sup>22-23</sup> Current work on OVD processes will attempt numerical modeling of what we believe may be an external burner with greatly improved thermophoretic efficiency. Preliminary experiments have shown this a possible solution to the low efficiency of OVD deposition during initial stages when the target rod is small. The numerical computations have been carried out on a Micro-Vax II, and will be continued on a newly acquired DEC Station 3100 as well as the super computer at the JVNC Center in Princeton. The codes we have been working with are based upon the SIMPLER algorithm, and have been FLUENT, from Creare, Hanover, N.H., and TEMPEST, a time dependent code from Battelle.

The concept of a "thermophoretic" nozzle is described in the following. In the fabrication of large boules for optical fibers, two types of torches for outside processes have been utilized, one for OVD (Outside Vapor Deposition), the Corning process, and one for VAD (Vapor Axial Deposition). In the OVD torch, the dopants flow through a central hole surrounded by premixed combustion gases. In the VAD torch, the reactants flow through a center tube, and in a series of annuli, oxidizer, inert gas, and fuel are transported. It has been shown recently, that the VAD torch used in OVD is more efficient than an OVD torch when the boule size has become an appreciable fraction of the VAD torch diameter.<sup>24</sup> In both the VAD and OVD processes, deposition rates of the order of 10-20 gm/min are considered large. However, in the initial stage of OVD, when the "bait rod" is quite small, deposition with torches designed for large mass transport is quite inefficient. We propose a simple modification of a VAD torch that will render it efficient during the initial stages of OVD deposition where the target rod is very small and that can be used with an aerosol and a silica transport approaching 1 gm/s.

In Figure 1 is shown a typical VAD torch. We place two parallel quartz, or ceramic plates as a subsonic converging nozzle in the path of the reacting jet as shown. This will focus the gas molecules. If there is a chemical reaction producing "soot", whether the particles impact upon the plates will depend upon the relative temperature of the gas stream and the plates. If these plates are at a higher temperature than the gas stream, heated either electrically or with external torches, then the "soot" particles formed will not impact upon the plates, but will be kept away by thermophoretic forces. This thermophoretic nozzle can

then be used to focus the output of a large VAD torch on a thin "bait rod" to increase deposition efficiency in the initial stages of the OVD process, which can take many hours. Preliminary experiments as well as numerical computations indicate that this is a promising solution to the problem of low deposition efficiency during the initial stages of an OVD fiber preform.

### 2.3 A Novel Fiber Device for the Measurement of Electric Field

Many techniques have been proposed for the measurement of electric field using photonic concepts.<sup>25-32</sup> We consider a novel technique by which electric field (AC, DC) may be measured using a combination of a Faraday cage and a Fabry-Perot cavity at the end of an optical fiber. The device can be fabricated using microelectronic processing. It is believed that this configuration can be made sensitive, reliable, and economical. It is also believed that the response time of this device can be made small enough to be of interest for large electromagnetic pulse measurements. The basic concept has been previously established with a related device that has been proposed and fabricated by AT&T Bell Laboratory, Murray Hill, for the purpose of making sensitive pressure measurements on an extremely small scale<sup>31-32</sup> and is described in the following.

The transmissivity (or reflectivity) of a Fabry-Perot cavity is extremely sensitive to changes in the length of the cavity that may be caused by thermal expansion, or in the case of a flexible mirror arrangement on the end of the cavity, by a pressure differential. Consider the fabrication of a Fabry-Perot cavity using microelectronic techniques, with dimensions as shown in Figure 2. If we imagine that the top surface of the cavity is an elastic membrane, and that the internal pressure of the cavity is sealed from the external pressure, then, a pressure differential across the top surface will cause a deflection of the membrane. This is given by the simple formula,<sup>33</sup>

$$\delta h(r) = \frac{R^4(\delta P)(1 - v^2)(1 - (r^2/R^2))}{16(t^3)E_y} \quad (1)$$

where  $\delta h(r)$  is the deflection of the membrane (the top reflecting surface of the Fabry-Perot),  $\delta P$  is the pressure differential across the membrane,  $R$  is the membrane radius,  $t$  is the membrane thickness,  $E_y$  is Young's modulus for the membrane, and  $v$  is Poisson's ratio. In the following, for ease of illustration, we will consider that the radius of the cavity  $R$  is much larger than the cavity length,  $l$ . This will permit us to consider a planar deflection of the cavity at its center,  $r=0$ .

If the Fabry-Perot is mounted on a fiber, as illustrated in Figure 2, then light coming into the Fabry-Perot will be reflected back along the fiber. The reflectivity of the Fabry-Perot will be a function of the cavity length that will depend on the external pressure as indicated in equation (1). The formula for the reflection of a Fabry-Perot cavity as a function of cavity length is given as follows:<sup>34</sup>

$$I_r/I_o = \frac{4\sqrt{R_1 R_2} \sin^2(\frac{2\pi h}{\lambda})}{(1 - \sqrt{R_1 R_2})^2 + 4\sqrt{R_1 R_2} \sin^2(\frac{2\pi h}{\lambda})} \quad (2)$$

where,  $R_1$  and  $R_2$  are the reflectivities of the cavity,  $h$  is the cavity length where  $h = h_0 - \delta h$ ,  $\lambda$  is the wavelength, and  $\delta h$  is given by equation 1. By changing the membrane stiffness, or its thickness, sensitivity over a wide range of pressures can be obtained. In the following we describe how the sensitivity of a Fabry-Perot cavity may be used in the measurement of voltage and electric field. This can be done using (essentially) dielectric materials that will not perturb the field in any macroscopic sense, and whose measurement will not be influenced by electrical noise. Using microelectronic techniques to fabricate a Fabry-Perot cavity that is either conducting, or coated with a conductor, then, if the length of the cavity is much smaller than its width, end effects may be neglected in a description of the electric field within the cavity. That is, we have constructed an ultra thin Faraday cage from which the external electric field is excluded. To first order, this will be equivalent to two conducting parallel plates with a field of magnitude  $E$  external to the cavity. As a consequence, there will be an external pressure on the surface of the cavity given by

$$\delta P(E) = \epsilon_0 E^2/2 \quad (3)$$

If this expression is substituted into the formula above for the deflection of the membrane, we obtain the deflection of the membrane at the end of the cavity as a function of electric field. It is necessary that the

physical properties of the cavity be chosen carefully if we wish to insure that there is a notable degree of linearity in the signal. For the two materials considered here, quartz and platinum, the cavity dimensions, i.e., radius, membrane thickness, and height, are all within the range that is readily accessible within present laboratory capabilities at both Brown and AT&T Bell Laboratory.

As a specific example, we consider a cavity of platinum that is  $100 \mu$  in diameter, the top reflecting membrane is  $.5 \mu$  thick, and the cavity height of  $3 \mu$ . By a proper choice of parameters, it is possible to achieve a monotonic variation of reflected signal intensity over almost any desired range of electric field. In the example chosen, we have a reflected intensity of 1.0 for an electric field of  $400 \text{ V/cm}$ , and a signal of .52 for an electric field of  $40,000 \text{ V/cm}$ . This is shown in Figure 3. Greater sensitivity can be obtained over smaller ranges, or a comparable change in signal can be obtained over a wider range of voltages. It should be noted that greater sensitivity may be obtained by the electric field sweeping the cavity through several free spectral ranges. This concept can be extended to measure all three components of the electric field vector. As a consequence of the extremely short length of the Fabry-Perot cavity, it should be insensitive to changes in temperature, which is unusual for interferometric sensors. Work is in progress in the fabrication of cavities of a similar design, but that will exhibit greater sensitivity to the electric field.

#### 2.4 An Automated Fused Taper Coupler Station

Fused taper couplers are fabricated by placing two fibers next to one another, heating, and pulling them, so that the evanescent wave of one core is coupled into the other. The specific success of such a process depends in large measure on a meticulous attention to all of the details of the process to guarantee that the pull of the fiber is uniform. C. Baldwin (Polaroid Corporation) had developed an automated fused taper coupler station in which all stages of the process are computer controlled with constant feedback to insure uniformity of coupler fabrication. We have recently received this equipment and are continuing a cooperative program of research with Polaroid with the goal of automating a process that will fabricate  $N \times N$  couplers. Usual techniques of coupler fabrication require that the fibers (traditionally  $125 \mu$  diameter) must be etched to allow the cores to come close to one another with a short fusion length. By using fibers of a smaller cladding diameter, but with the proper core for a single mode fiber, fused taper couplers may be obtained with consistency. These thinner fibers can then be fused onto a standard size fiber having the same core diameter. The goal of this aspect of our research would be to study how this coupler station could be modified to fabricate inexpensive  $16 \times 16$  star couplers and beam splitters.

Our program in developing "soft" glass fibers with high index that exhibit nonlinear properties will complement the work on fiber couplers. These couplers form the building blocks of various nonlinear passive optical elements.

#### 2.5 Embedded Fibers as a Diagnostic Tool for Composite Materials

As increased demands are placed upon the development of composite materials to be employed near the limits of new technologies, it becomes necessary to monitor their state with regard to temperature and strain in a continuous manner. A technique that holds promise of fulfilling this goal is that of embedding optical fiber sensors in the material as it is being processed. These fibers must be able to operate over the same range of temperature and strain as the host composite. By measuring the light passing through the fiber, it is possible to determine information on the state of the surrounding composite.

The basic physical principal of embedded fibers to characterize the state of a composite material is that light sent through the fiber has either its intensity, phase, or polarization altered by changes in the mechanical state of the surrounding host. This can ultimately be used to provide "real time" information on the state of a specific structure. However, in order to achieve this ultimate goal, a fundamental understanding of the nature of the fiber-host matrix interaction is needed. Optical fibers embedded in a fiber reinforced composite material can be used to study the constitutive behavior of the material and how the reinforcing fibers interact with the background matrix. This is the focus of this research. Only when such behavior is understood, can "smart skin" composites that monitor and control the state of the structure become a reality.

Many laboratories are engaged in research on this topic. Our emphasis will be on fiber design and fabrication to use embedded fibers as a diagnostic tool for fundamental studies on composite materials, as

opposed to composite structures. With few exceptions, fibers have thus far been designed for telecommunication, and, in spite of the rapidly growing sensor market, insufficient effort has been expended to optimize the fiber design for its specific sensing role. This question must be addressed if there is to be any hope of embedded fibers fulfilling their promise. As noted above, Brown is one of the very few universities capable of designing and fabricating all stages of state-of-the-art optical fibers. Fiber design with respect to novel dopants that can maximize the sensitivity to temperature and strain will be one emphasis of our program. It has been noted that certain rare earth elements, in particular Nd, exhibit a marked temperature sensitivity, and we will study the effects of dopant levels upon temperature sensitivity for embedded fibers. Since we will limit ourselves to consider maximum lengths of fiber of the order of a hundred meters or so, attenuation due to higher dopant levels will not be a limiting factor as would be in the case of the design of a telecommunications fiber.

One of the techniques used in the interrogation of a specific fiber section is that of OTDR (Optical Time Domain Reflectometer). In this technique, an optical signal of short duration is launched into an optical fiber. At each point in the fiber, there is some amount of backscatter from this pulse. The amount of backscatter is dependent upon the fiber design, geometry, the material with which the fiber is doped, the level of doping, and the local state of temperature and stress. Clearly this is a system with many parameters. Knowing the speed of propagation of the wave in the fiber, and knowing the time difference between the introduction of the pulse into the fiber and the time of the return of the back scattered wave, it is possible to obtain information on the local position from which the pulse was scattered. This back scattered pulse can provide information on temperature, strain, and other characteristics of the fiber locally.

One specific type of fiber design that would be of particular interest for OTDR experiments has been suggested by M. Kleinerman. In this fiber, a weakly guiding single mode core is surrounded by an annulus of a high numerical aperture multimode fiber. When the fiber is perturbed, the light leaks from the weakly guiding core into the multimode annulus. The speed of light in this annular wave guide will be different than in the core, so a detector will perceive two pulses at the end of the fiber, one from the core, and one from the surrounding annulus. The difference in arrival time of the two pulses will determine where the disturbance occurred, and the magnitude of the pulse in the multimode region will determine the magnitude of the disturbing parameter. Work is presently in progress to fabricate such a fiber.

Thus far, all experiments on the behavior of embedded fibers in composite materials have utilized whatever existing fibers may be available, and these have uniformly been either single or multimode telecommunications fibers developed for long line transmission at either  $1.3 \mu$  or  $1.55 \mu$ . Indeed, the whole question of the optimum configuration of a fiber for the sensing of a particular parameter, be it temperature, strain, or any other external physical perturbation, has yet to be addressed in any systematic manner. This will be one focus of our program.

### 2.5.1 Visible, Single-Mode, Polarization Maintaining Fibers

In the following we will attempt to outline the development of our program on a host of fibers uniquely suited to the experiments in composite materials research that have been outlined above. Since ultra-low losses are not a primary concern in sections of fiber of the order of tens or even hundreds of meters, there is a distinct advantage in performing composite materials-fiber research in the visible, where transmission losses associated with Rayleigh scattering will be higher than in the near infrared. Detectors are simpler, fast, and any interferometric techniques are more comfortable with a visible signal.

The sources for single mode fibers in the visible that are capable of maintaining single polarization are limited. Since strain effects fiber polarization, a sensor fiber with these characteristics would be advantageous in an embedded configuration in a composite material. The technique we plan to employ in obtaining these fibers for our composite materials project similar to that developed at the University of Southampton, England, and used in the York Technology "Bow-Tie" anisotropically stressed fibers. We have done preliminary experiments on internal  $\text{CO}_2$  laser etching of MCVD preforms, and will continue this to obtain polarization maintaining fibers in the visible, as well as at traditional telecommunications wavelengths.

### 2.5.2 Twin Core Fibers with Laser Etching Techniques

Twin core fibers have been used in a host of experiments to monitor strain in a fiber. As the fiber is stressed, the light from the evanescent wave in one core "leaks" into the neighboring core. Thus, integrated

strain can be obtained directly through an intensity measurement. These fibers are usually fabricated by cutting two preforms nearly in half, optically polishing the surfaces, and then placing them together in a draw tower furnace. This is a rather costly technique, and we propose to study the fabrication of such fibers using techniques of internal laser etching and deposition directly on our MCVD equipment. This is outlined in Figure 4. Work is in progress on this new type of "twin-core", or multicore fiber. It is also relatively easy to use a modified "rod-in-tube" technique to fabricate multicore fibers; however, if the core rods require special doping this may not be convenient.

### 2.5.3 OTDR (Optical Time Domain Reflectometry) Fibers

In Optical Time Domain Reflectometry (OTDR) a pulse of light is sent into the fiber. At each point along the fiber there is some back reflection, and where there are gradients in the local environment that influence the index of refraction of the fiber, the amount of this back reflection is increased. Many commercial OTDR's are presently available, and their main use is in the location of fiber imperfections, or of breaks in the fiber. The temporal width of the input laser pulse, and the accuracy with which the return reflection can be gated determines the spatial accuracy of the location from which the back scattered photons are emitted. In many cases, it is important to resolve the effect of a disturbance on a scale of the order of a centimeter. Picosecond technology has demonstrated this to be feasible.

Fibers are generally designed for their transmitting properties. In using an embedded fiber to measure the state of a composite material, a more highly backscattered signal would be desirable to increase the signal/noise of the return photons. This could be accomplished in several ways. The fiber could be doped with compounds in the core that retain a microcrystalline structure to enhance local backscattering, or local radiation induced color centers could be implanted in the fiber in a simple manner to increase back scattering. Part of this aspect of our program will be to specify the parameters for a sensing OTDR fiber.

### 2.5.4 Fiber with Increased Microbending Losses

Customary fiber design emphasizes the necessity of reducing losses due to bending in the fiber. Indeed, pressure sensors have been developed that make use of the degradation of the optical signal as a function of fiber bending. We have the capability of fabricating high numerical aperture, large core fibers that inherently will show large bending losses. In addition to this, we propose to study the effects of a soft coating on the fiber in which hard spherical particles are embedded during the coating process (of the order of ten  $\mu$  in diameter). With lateral force on the fiber these particles will serve as initiators of severe bending losses in the fiber. Such a fiber may be of use in the monitoring of strain in large composite plastic sections, such as is envisioned in future development of composite submarines, or similar structures.

Another type of application for an embedded fiber would be to locate instabilities that arise in the fabrication of composite materials, as layers of fibers are wrapped to provide strength members for the surrounding material. For thick epoxy-type composites, errors in the curing process can lead to instabilities in the curvature of the fibers embedded in the material, as shown in Figure 5. Here we observe a radius of curvature of the order of many centimeters, followed by a section in which the embedded fiber curvature is of the order of a few centimeters. It is at this location that subsequent failure will occur. We propose a simple technique for determining if there is a change in the radius of curvature, and for locating this critical radius.

By embedding an optical fiber in the composite material, or a series of fibers along with the strength producing fibers, we can send a light signal through the fiber. If the fiber suffers bending beyond a critical radius, there will be a large loss of energy in the transmitted light, and the intensity of the signal propagating through the fiber will decrease accordingly. This critical radius is given as follows.

$$R_c = \frac{3n_1^2 \lambda}{4\pi(n_1^2 - n_2^2)^{\frac{1}{2}}} \quad (4)$$

If the critical radius for which the composite is weakened is known, then this must be set equal to the critical radius  $R_c$  of the fiber. If the radius is larger than this, then there is little or no attenuation of the signal propagating through the composite material. If the radius is smaller, the losses will be large.

In order to determine the location of this bending flaw, usual techniques of OTDR (Optical Time Domain Reflectometry) may be employed.

### 2.5.5 Stress Induced Birefringence in Fibers

There are two ways in which the degeneracy of the solutions for Maxwell's equations can be broken in an optical fiber to produce birefringence. That is, waves traveling parallel and perpendicular to an axis will have different propagation speeds. This symmetry breaking can be done with either geometry, or local strain, or a combination of both. It can be shown that stress is the dominant effect in most polarization maintaining optical fibers. The average stress induced birefringence in a fiber is given as follows.

$$\bar{B} = \frac{-C \iint_{core} (\sigma_1 - \sigma_2) dA}{\iint_{core} dA} \quad (5)$$

where C is the stress-optic coefficient, and  $\sigma_1$  and  $\sigma_2$  are the principle stresses inside the core. Solutions for fiber birefringence are usually obtained by finite element calculations. However, for several geometries of importance, shown in Figure 6, an elliptical core, a twin core fiber, and a "Panda" fiber, K.S. Kim and K. Tsai have obtained simple closed form solutions for birefringence.<sup>35</sup> Inside the inclusion, the total stress distribution is the superposition of uniform stresses from Eshelby's solution<sup>36</sup> and the stresses derived from complex potential functions. These results for an elliptical core fiber are shown in Figure 7.

### 2.6 Electrically Polled Anisotropies in Optical Fibers

In this section we wish to discuss the concept of polling fibers in an electric field to enhance nonlinearities. Recent work at Southampton has shown that by drilling holes in a preform of the order of two millimeters (this corresponds to a typical core size in the MCVD preform) it is possible to maintain these axial holes in the fiber during the pulling process. Later insertion of electrodes into these holes has permitted the field to be of the order of several hundred volts/micron. This work was done to pursue work in second harmonic generation. We are initiating a program to study the doping of a core region with either a polarizable anion or cation. The glass will be raised to its transition temperature and the electric field will be turned on. In its quasi liquid state (albeit with a high viscosity), there will be a dipolar alignment with the electric field that will enhance the nonlinearity of the medium. The glass will be cooled with the field on, thus freezing in the alignment. Using the Nernst-Planck equation, and Poisson's equation, we have obtained numerical solutions of the dipole, anion, and cation distribution in the fiber as a consequence of the initial doping, field strength, diffusion coefficients. These calculations are being extended to include the rotational diffusion equation obtained by Debye. The ion distributions can be related to the refractive index, and thus, to the nonlinear properties of the optical fiber.

### 2.7 Rare Earth Glasses: Sol Gel Techniques

A program is in progress to develop sol-gel techniques to prepare glasses of interest for optical fibers. This work is directed by Prof. W. Risen, Department of Chemistry. Sol-gel methods have been developed for preparing homogeneous binary silicate glasses incorporating significant amounts of nearly all of the lanthanide ions (1), and actinide ions Th and U(2). These dried and densified glasses are x-ray and microprobe amorphous, and free of significant OH overtone and combination band absorption in the near-infrared. The glasses of the lanthanide series have now made available in binary silicates the wide range of electronic absorptions and magnetic susceptibilities known for these ions, which have attractive Faraday effect possibilities as well as important uses in fiber lasers. The actinide glasses were shown to form with the uranyl ( $UO_2^{2+}$ ) ion and with covalently bound Th, which impart polarizability contributions due both to the cations and to covalent-metal-oxygen bonds, needed for non-linear optical applications.

In the most recent period, this aspect of our program has been extended to the more complex problem of incorporating transition metal ions. Since such ions exhibit a wide range of oxidation states and coordination geometries. Previously reported work gave rather disparate results. Our goal was to incorporate ions of Cr, Ni and Mn, for example, in fully formed binary silicates and to determine their oxidation states. We have succeeded in preparing them with either Cr(III) or Cr(VI) with Ni(II), Mn(II), ions in well characterized

glasses and in very nearly dried gels treated at 700°C, homogeneous silicates with both Mn(II) and Mn(III) have been obtained. This work is being continued, using new chemical approaches and internal reductants.

There are four goals for this aspect of the program: to prepare new starting materials for fiber preforms; to develop analytical methods for fibers and preforms based on measured spectral properties of well characterized materials of desired compositions, and to develop new coating compositions. The preparation and spectral characterization of the binary silicates, together with careful study of the evolution of their structure during processing, directly addresses these goals.

Two different chemical approaches seem promising, since we are able to incorporate optically interesting ions via binary silicate gels. Both are designed to make it possible to apply films with high nonlinear index coefficients, at relatively low temperature, which can be processed by microelectronic techniques to form devices. In one, the indices in the regions of interest would be altered in processing by ion exchange, and in the other they would discover ways to incorporate at least two quite different ions in a silicate glass formed from a gel. Therefore, we will attempt to incorporate both an exchangeable ion, such as an alkali, and an ion which imparts a high non-linear index. We are presently attempting to make homogeneous gels and then glasses which contain  $\text{Cs}^+$  or  $\text{Na}^+$  together with  $\text{UO}_2^{2+}$ , or either of them with a transition metal oxide having both ion-oxygen covalent bonds and non-bridging Si-O bonds. A range of these compositions is needed so that optical as well as mechanical properties before and after exchange can be studied.

Other techniques for changing indices in small channels within high non-linear index silicate glasses involve two types of oxidation-reduction. In one, the added ion in exchange will have a larger charge than the one being removed so that the material making the principal contribution to the non-linearity, e.g., transition metal, lanthanide or actinide, can be reduced electronically without there being a structurally descriptive charge imbalance. For examples, exchange of  $\text{Ca}^{2+}$  for  $\text{K}^+$  in a material in which  $\text{Mn}^{3+}$  is reduced to  $\text{Mn}^{2+}$  (or,  $\text{Eu}^{3+}$  to  $\text{Eu}^{2+}$ , or  $\text{Co}^{3+}$  to  $\text{Co}^{2+}$ ), could accomplish this and lead to very interesting properties. We are working on ways to fabricate the ternary gels, produce homogeneous glasses and films, carry out the reactions, and then attempt the reactions through appropriate masks. The other oxidation-reduction approach will be to use highly reduced forms of incorporated materials to change the properties in exposed (channel) regions. For example, exposure of a region containing  $\text{Co}^{3+}$  or  $\text{Mn}^{3+}$  to  $\text{Co}_2(\text{CO})_8$  or  $\text{Mn}_2(\text{CO})_{10}$  vapors may reduce the incorporated metal ion, remove the excess oxide by forming  $\text{CO}_2$ , and leave a modified region without introduction of new species. In the case of certain such reactions, as with  $\text{Fe}(\text{CO})_5$ , photochemical assistance may be important. This introduces the possibility of using other masking techniques, and permit control of the rates of processing.

The process of forming glasses for preforms or other optical components, especially from gels, involves structural collapse or densification from disordered and higher free energy states. There a number of molecular scale processes involved, including chemical changes as well as physical ones. Among these processes, those associated with thermally activated rearrangements, related to general annealing, are of particular interest. We have found that large exotherms, not associated with crystallization or significant dehydration, can be observed in some new binary alkali silicates and that their magnitude and temperature is strongly related to their thermal histories. This may provide a way of measuring systematically the free energies and, perhaps, activation energies toward rearrangement of the hierarchy of disordered states. We are attempting to obtain such data on several series of binary silicates, and to place the interpretation on a firm basis. The initial studies have been done with DSC, TGA, x-ray and electron microprobe methods, but they will be extended using laser Raman and infrared microscopic reflectance methods in an attempt to determine the molecular properties which are changing through the process.

### 3. SUMMARY

The Laboratory for Lightwave Technology within the Division of Engineering at Brown University has facilities for the fabrication of optical fiber preforms, their characterization, and the drawing and characterization of fibers. We are engaged in a program of cooperative research with other universities and industry, as well as with other departments within the university. Our main focus has been on novel aerosol techniques for fiber and gradient index lens (GRIN) fabrication, sensors, in particular an electric field sensor, N x N fused taper couplers, and embedded fibers as a diagnostic device in composite materials. We are also concerned with specialty fibers that maximize optical nonlinearities and are beginning a program on electrically polling specially doped fibers to enhance nonlinear characteristics of the fiber. In the Department of Chemistry,

work is being carried out by Prof. W. Risen on sol-gel techniques for rare earth glasses. This complements our work in rare earth doping of fibers using aerosol techniques.

#### 4. ACKNOWLEDGEMENTS

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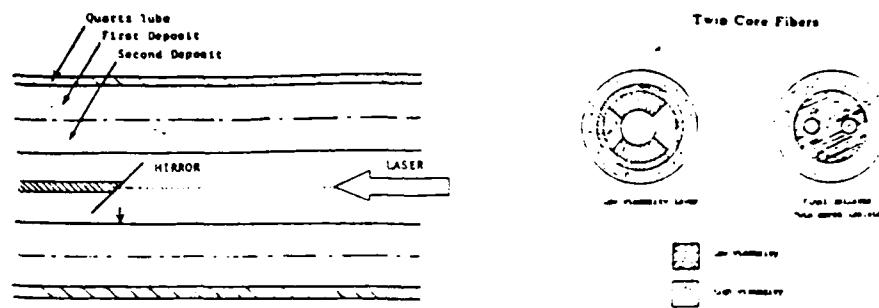


Figure 4. Laser Etching to Form Multicore Fibers

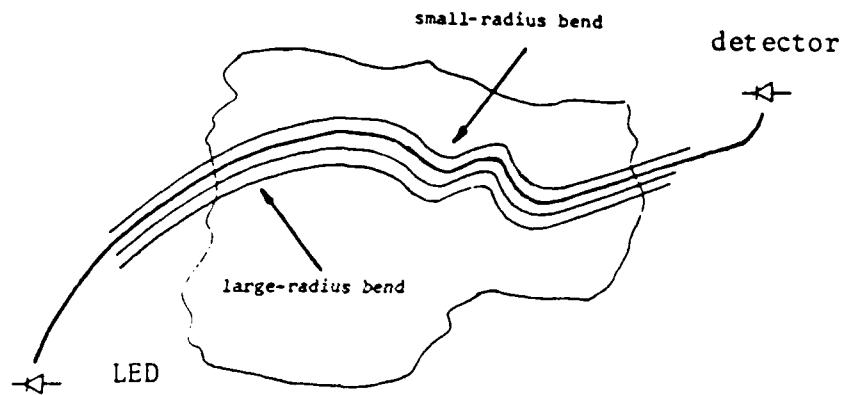


Figure 5. Fiber Detection of Composite Curing Instabilities

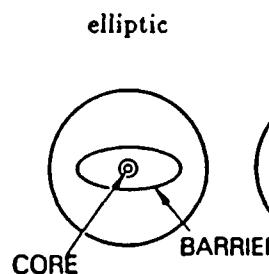


Figure 6. Birefringent Fibers

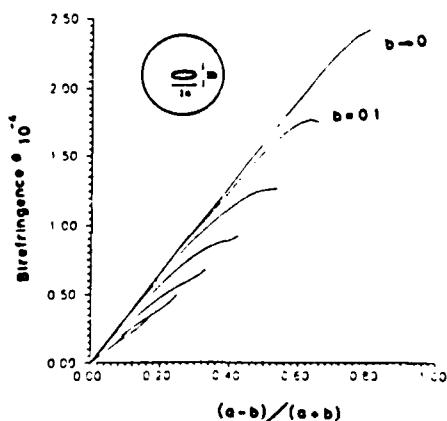


Figure 7. Birefringence of Elliptical Core Fiber

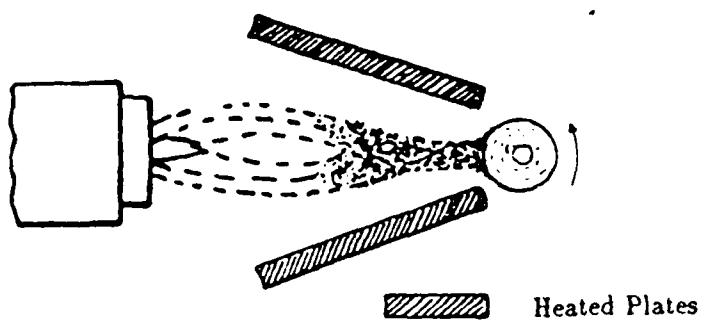


Figure 1. Thermophoretic Nozzle to Increase OVD Torch Efficiency

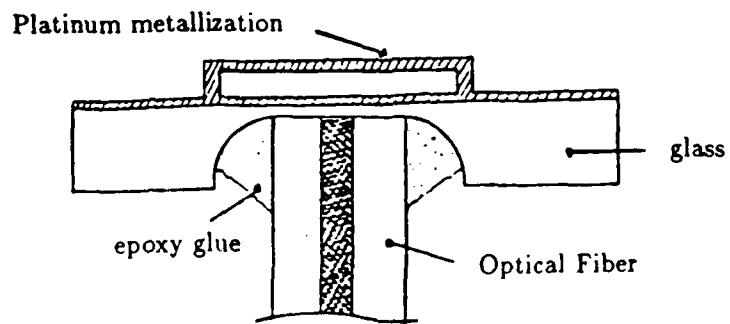


Figure 2. Mounting of Fabry-Perot on Optical Fiber

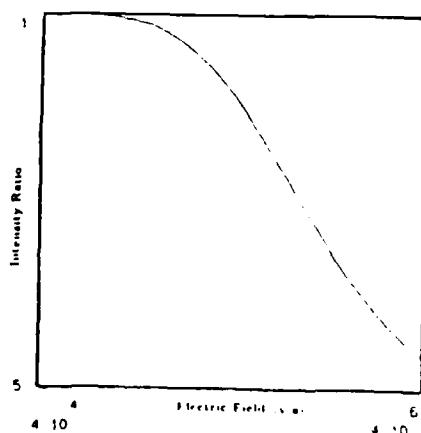


Figure 3. Response of Electric Field Sensor

## AEROSOL DOPING TECHNIQUE FOR MCVD AND OVD

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## ABSTRACT

Common techniques for the fabrication of optical waveguides (MCVD-Modified Chemical Vapor Deposition, OVD-Outside Vapor Deposition, VAD-Vapor Axial Deposition) depend upon the availability of high vapor pressure precursor compounds such as  $\text{SiCl}_4$ ,  $\text{GeCl}_4$ , and  $\text{POCl}_3$ . Vapor delivery techniques can not be used to transport compounds with a low vapor pressure. To incorporate such elements into the glass structure, we are investigating aerosol doping for both MCVD and OVD. Low mass flow rate aerosol transport is being used for core doping of rare earth elements in MCVD, and a high mass flow aerosol transport may have application in overcladding in OVD, the fabrication of fiber boules of glasses with a high nonlinear refractive index, and for GRIN (Gradient Index Lenses) lenses.

## 1. INTRODUCTION

The ease with which low loss optical fibers may be fabricated using various chemical vapor deposition processes (MCVD, OVD, VAD) is largely dependent on the availability of liquid precursors such as  $\text{SiCl}_4$ ,  $\text{GeCl}_4$ , and  $\text{POCl}_3$ . The transport of these vapors is effected by a carrier gas, usually oxygen, into a reaction zone where the desired glass oxides are formed at elevated temperature and then thermophoretically deposited on a suitable substrate. The deposition can be either internal, in the case of MCVD, or external, for OVD and VAD. For fibers used in the telecommunications industry, this "distillation" of the reaction precursors insures the ultra-high degree of purity required to insure that losses are mainly determined by Rayleigh scattering.

In MCVD the volatile chlorides of silicon, germanium, and phosphorus (typically) with oxygen as a carrier gas are introduced into a rotating tube on a glass lathe. These compounds are heated by an external torch to produce oxides in the form of submicron glassy aerosol particles. The particles are then deposited on the wall of the tube, largely through thermophoretic forces. See Figure 1. The torch passes over the deposited aerosol particles sintering them into a glassy, pore-free vitreous layer. After one pass of the torch, a thin layer of glass has been deposited along the tube. Different reactant flows with each pass of the torch permits a radial gradient in the index of refraction to be formed. Upon completion of the deposition, the tube is thermally collapsed to give a solid glass rod which can be pulled into an optical fiber.

There has been recent interest in the incorporation of dopants that can provide a host of lasing transitions in the near infrared, in particular, rare earth elements in the form of oxides. These are of possible use as amplifying sections in a telecommunications network as well as for certain specialized optical fiber sensors. An excellent review of fiber lasers is presented in reference (1). One standard technique in MCVD to incorporate these low vapor pressure compounds has been developed in the Electronics Department, University of Southampton, England. A section of the MCVD preform is expanded and coated with the rare-earth chloride.<sup>2</sup> This section is then heated to approximately 1100 °C to produce a rare earth chloride vapor which then passes into the reaction zone where it is oxidized and incorporated as a dopant into the silica based glass. With this technique, the amount of rare earth oxide that can be incorporated may be limited, and there may be undesired axial gradients in the composition of the deposited rare earth oxides. A second technique employs solution doping, in current use at British Telecom and other laboratories, where the preform is taken from the glass lathe and immersed in a dopant solution of either water or alcohol. The layers into which this solution penetrates are usually unsintered to increase dopant incorporation. Subsequent drying of the preform leaves a uniform distribution of the rare earth chloride which reacts in the flame zone to form

the oxide. In the case of OVD or VAD,<sup>3</sup> the soot boule is immersed in a doping solution and subsequently consolidated. Control over the radial distribution of the rare earth dopant with such a technique may be difficult. Another method, developed by Snitzer and Tummenelli at the Polaroid Corporation,<sup>4</sup> employs rare earth chelates that are heated to approximately two hundred degrees centigrade (depending upon the rare earth element) to insure an adequate mass transport into the reaction zone in MCVD. Patents have been issued involving aerosol transport for the doping of glass fiber preforms with alkalai metals, or members of the lanthanide family.<sup>5, 6</sup> In all of these proposed aerosol techniques,  $SiCl_4$  is used as the glass forming precursor. This prevents, for example, the efficient incorporation of some alkalai oxides since the chloride may be more stable than the oxide.

In the work to be reported here we have used an aerosol doping technique, which consists essentially of nebulizing a solution of the dopant in an appropriate solvent and flushing this aerosol into the MCVD substrate tube (along with the usual vapor precursors) or into the OVD or VAD torch for use in the outside processes. Our aerosol deposition work is separated into two categories. In the first, we consider low mass transport for MCVD core doping, and in the second, high deposition rate transport for OVD and the formation of bulk glass. This aerosol doping technique may also have application for the fabrication of fibers with higher nonlinear refractive indices, as well as for GRIN lenses.

## 2. AEROSOL DOPING IN MCVD

For low deposition rate doping of the core in MCVD, we have used a modified "room humidifier" as the source of our aerosol. This has a 1.5 MHz transducer capable of producing a reasonably monodispersed distribution of aerosol particles in the range of several microns. Particles of this size are not influenced by gravitational settling, and they may be transported relatively large distances with uniform, laminar convection. In Figure 2 we see the rotating seal arrangement by which the aerosol is transported into the flame region in MCVD. The 1.2 cm tube (internal diameter) projecting no closer than 10 cm from the closest approach distance from the torch is surrounded by the flow of  $SiCl_4$ ,  $GeCl_4$ , and  $POCl_3$ . When the MCVD lathe is in the horizontal position, there are significant secondary flows driven by the buoyancy of the hot gas in the flame region. This is a factor in the uniform mixing of the reactants. Our low mass transport generator was capable of delivering 1 gm/m of aerosol, which appears sufficient for core doping of MCVD preforms. With this technique, we have obtained an incorporation of neodymium oxide of 6% by weight. We are still encountering problems with microcrystallinity. We believe this may be explained as follows. When the aerosol containing neodymium chloride dissolved in water, for example, is transported toward the reaction zone, the water evaporates to produce a submicron particle of the neodymium chloride that reacts with the excess oxygen to form neodymium oxide. This can result in small pockets of phase separation in the glass between the silica and the neodymium oxide. Work is in progress to use an aerosol of  $SiCl_4$ ,  $POCl_3$  and dopants in solution in the form of metal-organic compounds. By nebulizing an aqueous solution of neodymium chloride we have shown that axial uniformity obtained with this technique is relatively good. See Figure 3. The fibers drawn from preforms prepared in this manner exhibit typical absorption spectra of the neodymium ion, and work is continuing in this area.

## 3. METAL-ORGANIC AEROSOLS FOR MCVD

Some of the compounds described below are of interest because of their solubility in non-polar organic liquids, and because they reduce amount of hydrogen introduced into the reaction zone in the MCVD aerosol process. The hydrogen atoms can be removed by a reaction with chlorine, although in the case of fiber lasers shorter lengths are used than in telecommunication fibers and higher absorption/length may be tolerated. We have found that the the acetylacetones of neodymium and aluminum are readily dissolved in  $CCl_4$ . The resulting solution has a lower viscosity than water and is easily nebulized. The only source of hydrogen is then from the organometallic rare earth compound, which is scavenged by the chlorine of the reaction. Excess  $O_2$  in the reaction zone combines with C to form  $CO_2$ .

The general group of organometallic compounds we have nebulized is the rare earth salts of the 1,3-diketonates which have been known since the turn of the century.<sup>7</sup> More recently, research has been carried out with higher derivates of the  $\beta$ -diketonates.<sup>8</sup> These are compounds in which the end methyl groups of 2,4-pentanedione are replaced by larger alkyl- or aryl- groups. These groups can be fluorinated, further

reducing the amount of hydrogen. These are among the few rare-earth compounds which have been found reasonably volatile.

Two rare earth compounds of the  $\beta$ -diketonates are of special interest: a rare earth complex of 2,4-pentanedione (acac) and 1,1,1,6,6,6-hexafluoro-2,4-pentanedione (hfa). The compounds are easily prepared from a neutral rare earth salt, usually the rare earth nitrate and the  $\beta$ -diketonate. This is done in a solution of a water-alcohol mixture with the addition of sodium hydroxide to adjust the pH of the solution to about five. In both cases the hydrated  $M(acac)_3$  or  $M(hfa)_3$  is obtained, where "M" = e.g. praseodymium, neodymium, erbium, holmium or terbium. These products can be vacuum dried at elevated temperature. The acac-compound is less expensive since the hfa is more difficult to produce. The fluorinated compound has the advantage of containing fewer hydrogen atoms, where six of them are replaced by fluorine atoms. We were able to dissolve this product in tetrachloroethylene, which is a common organic solvent and has a reasonably low vapor pressure at room temperature. In addition, the surface tension is very low, which makes it convenient to form an aerosol with our 1.5 MHz transducer. By introducing the mist of the nebulized solution into the preform and decomposing the compounds, we have been able to deposit rare earth oxides in MCVD with  $SiO_2$ ,  $P_2O_5$ , and  $GeO_2$ . At the same time the hydrogen in the reaction is removed, because tetrachloroethylene decomposes to give off chlorine that scavenges the hydrogen. It has been shown that the addition of aluminum oxide of 4 to 7 mol% in a silica glass host makes it much easier to incorporate rare earth ions into a silica glass matrix.<sup>9</sup> Aluminum also forms compounds of the  $\beta$ -diketonates, which are soluble in our solvent, so that it is easy to add these to the solution to introduce aluminum ions into the preform. This has the effect of "expanding" the silica matrix and permitting increased amounts of other dopants before the onset of phase separation. We have thus far limited most of our studies of doping to neodymium, but the method is clearly applicable to the other rare earths, and work is in progress in this area.

Since the aerosol now contains possibly corrosive liquids that must be atomized, we have found it useful to separate the transducer of our low mass flow rate ultrasonic generator from these liquids, particularly since it was not designed to handle corrosive liquids. Our present design is illustrated in Figure 4, in which cooled water circulates in the chamber above the transducer and is separated from the liquid to be nebulized by a thin teflon membrane. If the water above the transducer is chilled as well as the solvent, the warmer temperature of the tube transporting the aerosol and the silica substrate tube prevent any accretion of aerosol on the tube wall. This occurs because thermophoresis can be effective on the small size aerosols formed with a 1.5 MHz transducer.

We have cited some problems with phase separation that we believe is caused by the fact that the aerosol produces dopant particles whose concentration may lead to phase separation. For this reason, it is desirable to insure that the aerosol contains both Si and the dopant to minimize phase separation caused by localized dopant concentration. We are beginning a series of experiments in which organometallic compounds containing the lanthanide are dissolved in  $SiCl_4$  to form the aerosol. The use of TEOS (tetraethylorthosilicate) described below is of questionable use in an internal process, because of the danger of an explosion within the preform tube.

#### 4. AEROSOL DOPING IN OVD

The collapse temperature of the silica substrate tube in MCVD (2100 °C) makes it difficult to form glasses with significantly lower processing temperatures than silica. This problem does not exist with OVD, since there is no substrate tube. High index, "soft" glasses are of interest as fibers for a host of reasons. It has been speculated that nonlinearities in high index glasses might be used in fibers to form switching circuits in an optical computer with linear architecture, as well as in such devices as nonlinear Mach-Zehnder interferometers.<sup>10</sup> Theoretical work using nonlinear fibers in a Fabry-Perot configuration indicate some surprising results on soliton propagation.<sup>11</sup>

These nonlinearities are electronic in origin and may be less than  $10^{-13}$  seconds. They are characterized by  $\chi^{(3)}$ , or  $n_2$ , where the nonlinear refractive index,  $n_2$ , is given by:  $n = n_0 + n_2 I$ .<sup>12</sup> Fused quartz has a small value of the nonlinear coefficient; however, recent work on a silica, niobium and titanium glass (20 % mole fraction of the latter elements) has shown an increase in  $\chi^{(2)}$  by a factor of two.<sup>13-15</sup> Highly polarizable ions such as  $Pb^{2+}$ ,  $Nb^{5+}$ ,  $Ti^{4+}$ , and  $Bi^{3+}$  are of particular interest for the applications cited above. In a fiber geometry, even though the nonlinear coefficients for most glasses are small, high intensities can occur when laser light is concentrated in the core of a single mode fiber. Further, although the nonlinear component of

the refractive index has been measured, many of these glasses have not been fabricated into waveguides, and only a rod-in-tube fabrication technique is available.

We have used the aerosol delivery technique in MCVD described above in an attempt to fabricate a Pb doped silica fiber. Lead oxide is of interest not only for its large value of  $\chi^{(3)}$ , but because of its possible use as a gradient index lens. Indeed, Vogel, et al.<sup>14</sup> have recently commented upon an inability to obtain a Pb doped wave guide, although the group at Polaroid has had some success by using thick walled substrate tubes and subsequently pulling into fiber with an open, PbO doped core in the preform.<sup>16</sup> Other work by Sumitomo using the VAD technique has resulted in a fiber with some Pb incorporation. Our initial effort using an aerosol delivery system for the incorporation of Pb into an MCVD fiber was not successful as a consequence of the fact that the boiling point of PbO is lower than the collapse temperature of the silica substrate tube.

These considerations have led us to a modification of the low mass flow dopant system developed for MCVD and to develop a suitable high mass flow aerosol-burner design for OVD. Our first aerosol delivery system was a modified room humidifier, which proved suitable for core doping in MCVD. The mass transport was on the order of 1 gm/min of aerosol. If we wish to fabricate bulk glass with an outside deposition process rather than dope the core of an MCVD preform, significantly higher rates of deposition are required. To achieve this goal, we have recently acquired a commercial aerosol generator that is able to deliver 100 gm/min of a TEOS aerosol. This corresponds to a silicon dioxide transport rate of 30 gm/min using TEOS, and with tetramethylorthosilicate this would be increased to 40 gm/min of silicon dioxide. This rate is high enough so that we may consider this technique applicable to fiber boules and GRIN lenses. The aerosol is produced with a 60 kHz transducer with a mean droplet size of 28  $\mu$ . There are some larger droplets produced, and gravitational effects are present. It is therefore necessary to design a torch that insures complete vaporization before gravity can change the aerosol trajectories in any significant manner. This has been accomplished, and we are improving our design with a thermophoretic nozzle (discussed below) to focus the aerosol particles.

## 5. METAL-ORGANIC AEROSOLS FOR OVD

We now consider some of the chemistry needed to achieve our desired combination of glasses with aerosol delivery. Our experiments using an aerosol in an OVD configuration are of a preliminary nature, but we have found that using TEOS in the form of an aerosol injected into either an oxy-hydrogen or oxygen-methane flame has produced uniform layers of silica. We have also shown that a mixture of TEOS, alcohol, barium and sodium ethoxides can form an aerosol and the reaction products produce a low melting point glass. In one case, sintering on the OVD rod occurred immediately at a temperature of 950 °C. Of importance in the use of TEOS is the absence of chlorine. This means that many of the components of optical glasses (Na, Ba, K, etc.) can be incorporated into a glass, and the index can be structured in a predetermined manner. This can not be effectively done with SiCl<sub>4</sub> as a precursor, since the reaction rates may favor the formation of the chloride of the dopant rather than the oxide. In addition, the diffusion coefficient of these elements in glass is high, permitting the possibility of deposition of layers of different refractive index whose variations may be smoothed during proper thermal processing. This is of key importance in the fabrication of GRIN lenses using OVD techniques. The diffusion of germanium, and even phosphorus is too slow to permit quality GRIN lenses with either MCVD or OVD even though there has been some effort in this direction. We note that our work in this area is of a preliminary nature, and that the glasses that we have produced have been of poor quality. Thus far we have merely demonstrated that low melting point glasses may be formed with an aerosol technique.

The above comments describe the motivation for our interest in the development of fibers whose nonlinearities are larger than those of a silica glass. The advantage of a TEOS aerosol is that many constituents of optical glasses, and high index glasses form alkoxide compounds that are soluble in alcohol and miscible with TEOS. Thus, all of the glass forming elements are in the aerosol. We should also note that TEOS can be obtained in optical grade at a price not too different from that of optically pure silicon tetrachloride.

## 6. SOME DESIGN CONSIDERATIONS FOR AN OVD-AEROSOL BURNER

Our initial design was a modification of a VAD torch. It has been shown that a VAD torch may be used efficiently in OVD deposition.<sup>17</sup> An early experiment in our laboratory used a modified VAD torch with our MCVD aerosol generator. The modification consisted of a 1.3 cm inner tube in the VAD burner

that was used for the TEOS aerosol transport. Previous experience with a 1.5 MHz transducer in MCVD showed the ease with which small aerosol particles may be transported into a reaction zone. The only disadvantage of this system was the small mass transport rate. With our new commercial aerosol generator, the mean particle size was  $28 \mu$  with some particles ranging up to  $70 \mu$ . Particles of this diameter are strongly influenced by gravitational settling. The effects of gravity on larger particles from our nebulizer can be easily observed if the particles travel further than roughly three centimeters from the nozzle exit where the aerosol is generated. Our VAD torch worked well when the aerosol generator with the 1.5 MHz transducer was used, since the aerosol particles could be transported along the inner VAD tube without condensation or deposition on the tube wall. This was not the case for the heavier particle formed with the 60 kHz transducer, and there was significant wall condensation of the TEOS aerosol on the inner tube wall. Only 20% of the aerosol was transported into the combustion zone.

We have recently designed a new OVD nozzle for aerosol generation that consists of a small annular flow of He around the tip of the aerosol generator to cool it and to prevent the flame from propagating to the nebulizer itself. Surrounding this are two slit nozzles with a high flow rate of oxygen (15 l/min) to guarantee that there will be complete combustion of the aerosol and that there will be carbon dioxide and not carbon as a reaction product. Although the TEOS burns, with the flow rate and size of aerosol used here, it will not sustain combustion without an additional heat source. Even a small flame of a few mm in height interacting with the aerosol and oxygen is sufficient to result in complete combustion.

We have spent some effort in developing a "thermophoretic nozzle" that will permit the efficient use of a large VAD torch with even a small OVD boule. If heated flat plates are placed in the flow field as indicated in Figure 5 at a higher temperature than the local flow, particles will not adhere to the plates. The particles can thus be focussed onto a smaller target, thus increasing the efficiency of deposition. Preliminary experiments and theory indicate this to be a promising avenue of investigation.

## 7. CONCLUSIONS

The present conclusions of this aspect of our program lead us to the following:

1. High flow rate of chemical reactants into the reaction zone with aerosol transport. Rates of 30-40 gm/min of silica may be obtainable. The combustion of the reactants appears complete. This technique may be applicable to overcladding MCVD boules or increasing deposition in the OVD process.
2. In the chemical reaction of TEOS that produces silica, there is no chlorine as a reaction product, so that scrubbing the effluent is not necessary. The reaction products are the desired oxides, carbon dioxide and water. Elimination of chlorine in the reaction products favors the incorporation of many dopants that modify glass structure.
3. By creating an aerosol that contains the host glass as well as the dopant (or glass modifier), we believe that uniformity can be obtained on a microscopic level, and "pockets" of microcrystallization can be avoided.
4. By continuously changing the mixtures of TEOS and glass modifiers flowing into the aerosol generator, it is possible to change the composition of the glass in a continuous, controlled manner.
5. This technique is also suited for the fabrication of large gradient index lenses (GRIN).

## 8. ACKNOWLEDGEMENTS

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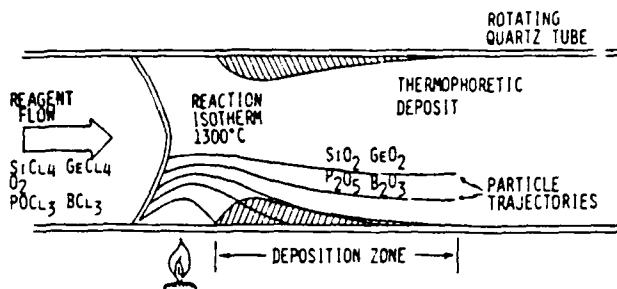


Figure 1. Aerosol Transport in MCVD

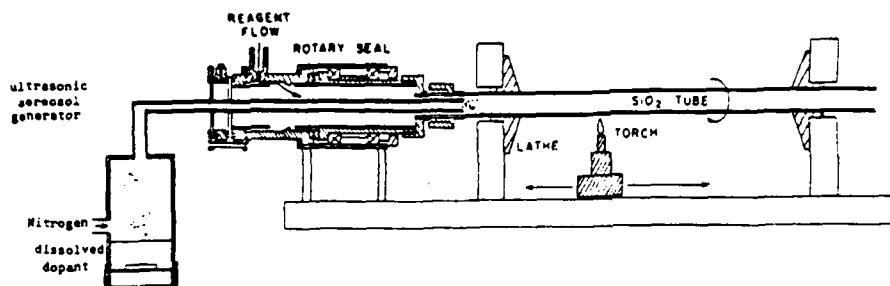


Figure 2. Rotating Seal in MCVD Aerosol Delivery

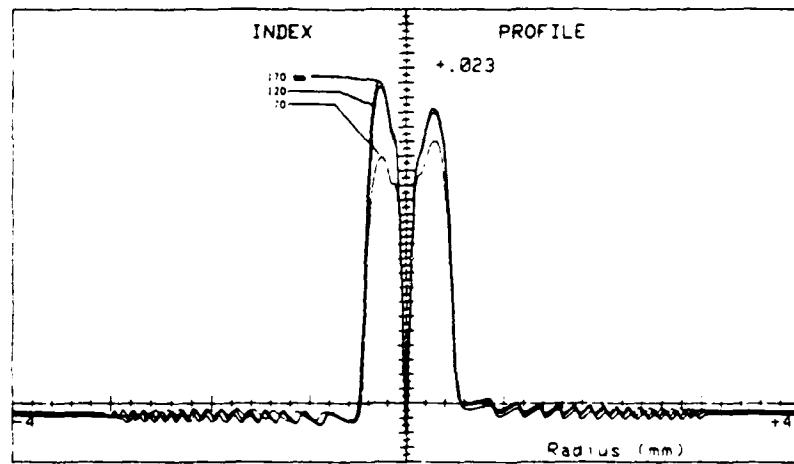


Figure 3. Axial Uniformity of Nd Doped Preform

NORSE

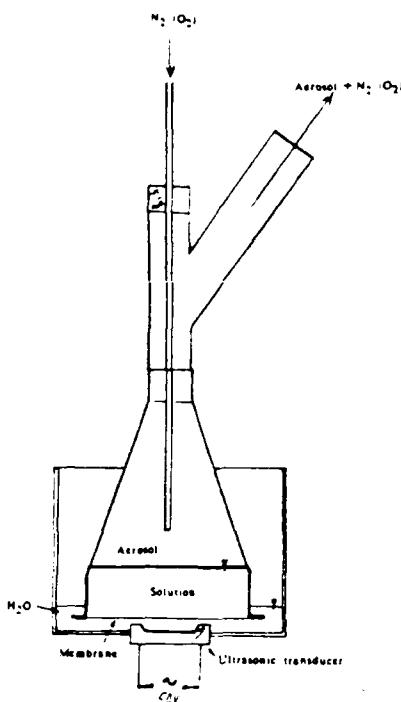


Figure 4. Design of Nebulizer for Corrosive Liquids in MCVD

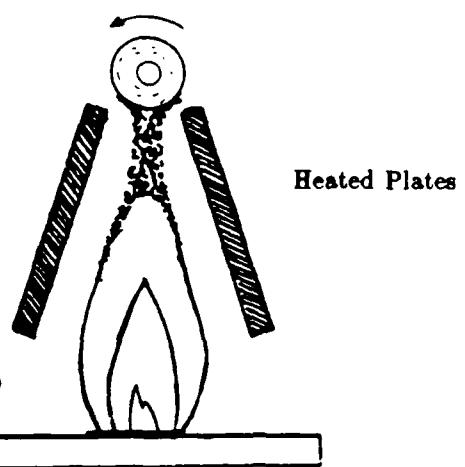


Figure 5. Burner Design for High Deposition Aerosol in OVD

